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AMERICAN
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VOLUME 41 AUGUST 18, 1952 NUMBER 17

SIZES FOR GLASS TEXTILES FOR REINFORCING POLYESTER PLASTICS

L P BIEFELD and T E PHILIPPS

Owens-Corning Fiberglas Corporation

In this paper the authors give: (a) an outline of the processing operations in which molten glass is converted into fibrous forms used for reinforcing plastics, (b) the requirements of a size which give satisfactory handling and performance characteristics to the fibers, (c) procedures for evaluating a size and (d) a comparison of the handling and performance characteristics of glass fibers which are surface-treated with various sizes.

INTRODUCTION

ADHESION between fibrous glass and plastics is an important requirement of high strength glass-fiber-reinforced plastics. The adhesion must be strong and not appreciably weakened during the use of the reinforced plastic part. It should not be greatly reduced by prolonged exposure of the part to water. If the adhesion is weak, and external forces are applied to the part, failure will occur due to poor stress distribution. The stresses will not be transferred from fiber to fiber through the low modulus, low strength plastic matrix. Thus, the reinforcing action of the high modulus, high strength fibers will not be realized.

Good adhesion is obtained by the use of coupling agents, which react with—— or bond strongly to——the glass fiber surface and to the plastic matrix. The bonds between the coupling agent and the glass, and those between the coupling agent and the plastic, must not be weakened by the constituents of the environment to which the glass-plastic part may be exposed. The most common of these constituents are water, weak acids and weak bases.

Surface treatments are used to apply coupling agents to fibrous glass. A material applied to the glass-fiber surface as it is formed is called a "size". A surface treatment given to the glass fibers after they are fabricated into strands, yarns or woven fabrics is called a "finish".

Surface treatment of the fiber as it is formed is preferred to surface treatment of the fiber after fabrication, since a freshly formed fiber has the highest strength and the most reactive surface. A good size will protect the fiber surface and thus cause the fiber to maintain most of its original strength. A coupling agent applied as an ingredient of the size will bond best to the glass-fiber surface in its most reactive condition.

The application of the coupling agent during fiber formation is also preferred because of the elimination of the subsequent extra steps of the finishing operation. The only reason for use of finishes at the present time is that no suitable size has been found which enables the fibers to be processed economically through the textile operations of twisting, plying and weaving, and which also enables the fabricated fibrous glass product to give good reinforcement to plastics.

Since the preferred application of surface-treating-agents is during the formation of the fiber, this paper is concerned principally with sizes. As the greatest interest at present is in the reinforcement of polyester plastics and, as different types of plastics require different types of coupling agents for best results, we shall confine ourselves to the discussion of sizes for fibrous glass to be used in reinforcing polyester plastics.

A size must impart two sets of properties to the fibrous glass. *First*, it must enable the fibers to go through the

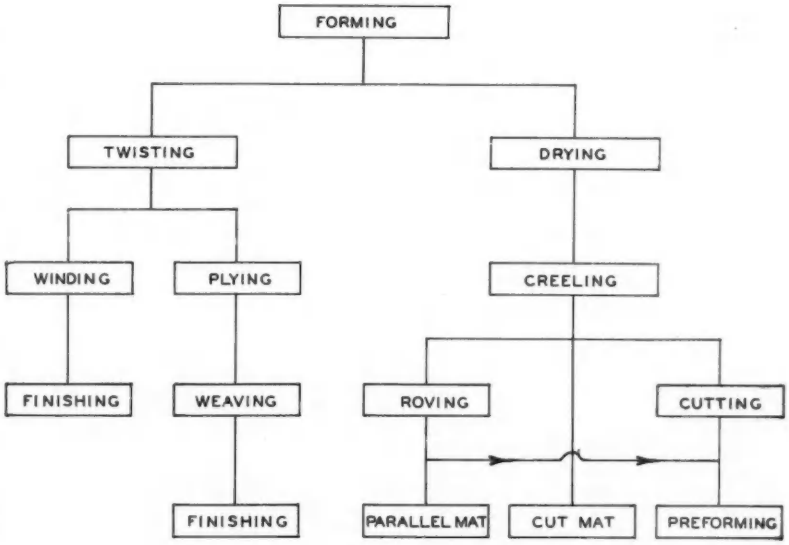
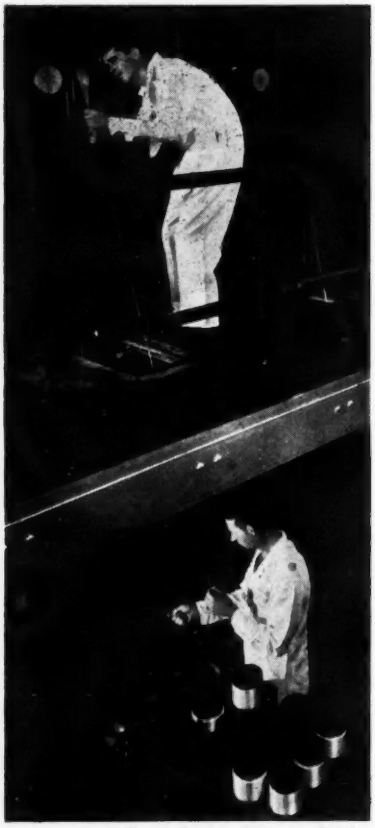


Figure 1
Flow Diagram of Textile Processing Operations



(Owens-Corning Fiberglas Corp)

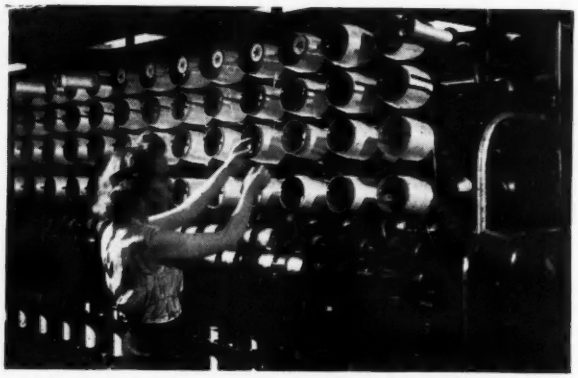
Figure 2
Forming Operation
Surface Treatments for Fibrous Glass

processing operations necessary to convert molten glass into fibrous glass in forms suitable for reinforcing plastics. This set of fiber properties is called "handling characteristics". *Second*, it must enable the fibers to reinforce the plastic in such a manner that the properties of the final glass-fiber-reinforced plastic are satisfactory. This set of fiber properties is called "performance characteristics".

PROCESSING OPERATIONS

An outline of the processing operations by which molten glass is converted into fibrous forms suitable for use in reinforcing plastics is best given by a flow diagram (Figure 1).

A photograph of the forming operation is shown in Figure 2. In this operation, glass is melted in a small electric furnace. The molten glass flows through orifices of a small bushing in the base of the furnace and is attenuated into filaments by mechanical drawing at very high speeds, normally, in the range of 5,000 to 10,000 feet per minute. Depending upon this speed and other variables, filaments having diameters from 23 x 10^{-5} to 75 x 10^{-5} inch are produced.



(Owens-Corning Fiberglas Corp)

Figure 3
Twisting Operation
Surface Treatments for Fibrous Glass

The surface treating material (size) is applied to the group of filaments, as they are drawn, between the bushing and the drawing apparatus. The size is usually wiped or sprayed onto the filaments at the collecting device, which gathers the filaments into a bundle known as a "strand". Below the gathering device is the "wind-up" apparatus, which winds the unit strand into a "forming package". All this takes place as the filaments are moving at the high rate of speed.

The forming package may be placed on a standard textile twister frame, the strand unwound and twisted onto a standard textile "twister bobbin". See Figure 3.

The twisted strands may be unwound from the twister bobbins onto multiple-wound packages. The fibrous glass in this form is given a finish and is then used to reinforce plastics, especially in rod form.

The twisted strands may also be plied into a yarn, which if desired, may then be woven into fabric. The fibrous glass in this form is also given a finish and used to reinforce plastics.

Forming packages may be dried under appropriate conditions and then placed on a creel. The strands from these forming packages are either wound together into a multiple wound roving package (see Figure 4), fed through a cutter, or fed into a "cut-strand-mat" forming machine. The roving package is a convenient package for handling and shipping glass in the strand form for use in preforming and rod fabrication. It is also a convenient source of strands for fabrication of "parallel strand mat" in an operation similar to warping.

SIZE REQUIREMENTS

The requirements of a size are determined by the ultimate properties the fibers must have to give good handling characteristics in the processing operations and performance characteristics in the use of the glass-fiber-reinforced plastic part. These requirements are best summarized by first considering each processing operation, and then considering the properties desired in the finished part.

FORMING———The forming operation requires that the size have low viscosity so that it flows readily through the applicating system, over the applicator and onto the filaments. The size must readily, uniformly and completely wet out the surfaces of the applicator and filaments. It should be stable to agitation, pumping and circulation. The size ingredients must not separate out in the size-applicating system, nor build up on the applicator or the traverse because of poor wetting, poor lubricity, poor stability, or evaporation of the dispersing medium of the size. The size must have sufficient lubricity to allow the filaments to pass at very high speed over the applicator and through the traversing mechanism that guides the strand onto the forming package. It must not be hazardous to personnel and equipment.

The combination of high speed drawing and rapid traversing of the strand on the forming package results in a whipping action of the strand, which sprays off a portion of the size into the atmosphere. Thus, the size must not be toxic, corrosive, inflammable nor cause skin irritation. It must bond the filaments into a unit strand but not bond the strands to each other on the forming package.

TWISTING———The strands in the twisting operation should have high run off and material efficiencies. The salvage, i e, the strand which cannot be readily unwound from the forming package, must be low. As the strand is twisted off the package, it must not form ringers, which are caused by filaments from the

original strand being held back by the other strands. These filaments strip away from the original strand as it is removed. The twisted strand must be free of "fuzz", filament ends which result from breaking of filaments by the guides and traverses in the twisting mechanism. All these handling characteristics of the strand are the result of the filament-to-filament bonding by the film-forming constituent in the size, the abrasion protection by the film-forming and lubricating constituents, and the softness due to a combination of the film flexibility and the ingredient in the size that acts as a softener.

PLYING———The plying operation is a twisting operation in which two or more twisted strands are plied together into a yarn, the size requirements for which are the same as for twisting.

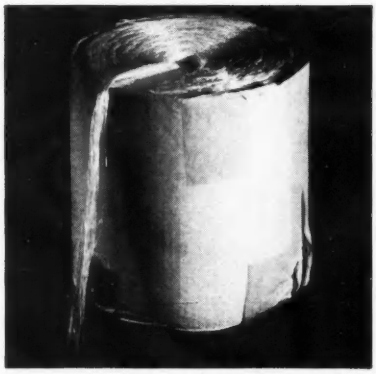
WINDING———For the winding operation, the yarn must be soft but not slippery. Thus, the film-forming, filament-bonding constituents must form a flexible film. The concentration of oil and glass lubricant in the size must not be too high.

WEAVING———The weaving operation requires a soft, lubricated and non-fuzzy yarn of good abrasion resistance. The size must have a softener, a glass lubricant and a good, flexible film-forming agent to give these properties to the yarn.

DRYING———In the drying operation, the conditions of drying should be suitable for the type of size on the fibers. The size should dry out so that no migration, discoloration or strand-to-strand adhesion takes place. The only constituent of the size that may be appreciably volatile under the drying conditions is the dispersing medium, which is usually water.

ROVING———After drying, the forming packages are set up on a creel for the roving operation. The size should lubricate the filaments sufficiently so that they do not drag or break up as the strand passes through guides, tension devices and the package traverse. The filaments must not be lubricated too well in order that a good package build-up may be obtained. The size must form a flexible, tough film to give the filaments an abrasion-resistant coating and must prevent high static charge build-up on the strand as it goes through the operation. The film must cause the strand to maintain its unity and to pull out of the forming packages, thus obtaining good run-off efficiency.

CUTTING———If cut strands or cut-strand-mat are desired, the dried forming



(Owens-Corning Fiberglas Corp)

Figure 4
Roving Package
Surface Treatments for Fibrous Glass

packages are set up on a creel and the strands fed through the cutting apparatus. In these operations, as in the roving operation, the size must impart sufficient lubricity to the fibers in order that drag and filament breakage may be minimized. Excessive filament breakage causes fuzzy strands and fly. The lubricity must not be too great nor the strand too flexible or soft so that the fibers are readily cut. The film-forming, filament-bonding constituent of the size must maintain strand integrity, but some opening up of the strand is desirable. The right amount of opening up of the cut strand is necessary for the felting of the strands as they are laid down in the formation of the cut-strand-mat or preform. If the strands do not open up, an open mat or preform having a large proportion of voids will result. If the strands open up too much, the resulting mat or preform will be fuzzy and will contain a large number of fuzz balls, i e, clumps of filaments. The mat or preform will be nonuniform and difficult to impregnate.

The size requirements for good handling characteristics of the fibers may be briefly reviewed as follows:

The dispersion medium of the size should be water; the size must contain a tough, flexible film-forming constituent to bond the filaments together in a unit pliable strand, and to give the filaments abrasion resistance; the size must contain a glass fiber lubricant so that the fibers move freely over surfaces contacted during pocessing; for twisting, plying and weaving, the fibers must have a size which gives greater lubricity, abrasion resistance and pliability to the strand than is required or desired for a size applied to fibers that will be used for roving, cutting, preforming and cut-strand-mat formation; the size ingredients must be so balanced that the strand has the right degree of integrity, pliability, lubricity and high abrasion resistance.

In order to impart good performance characteristics to the glass fibers, the size must contain a coupling agent which gives good adhesion between the fibers and the plastic; the adhesion must be maintained even in the presence of water. A glass lubricant must be in the size to give the fibers good handling characteristics; but it must not be so powerful in lubricating action that the glass-to-plastic adhesion is impaired. The film-forming ingredient, which must be present in the size for good handling characteristics, must be compatible with the plastic. It should adhere well to the plastic even in the presence of water. The film should not be destroyed by water or moisture. This size ingredient should not bind the filaments together too tightly, but should allow some end-separation during cutting so that rapid penetration of the plastic into the strand and complete wetting of the filments in the interior of the strand is obtained. The fibrous glass should be wet out rapidly and completely by the plastic during impregnation to achieve good adhesion.

Thus, for good performance characteristics, the essential requirement of a size is to promote good adhesion between the glass fibers and the plastic. The constituents other than adhesion promoters in the size are for imparting handling characteristics rather than performance characteristics. This is the reason that only a coupling agent is applied as a finish to fabricated fibrous forms for reinforcing plastics. However, a combination of film-forming material, a glass lubricant and a coupling agent must be applied in a size to the glass fibers as they are formed so that they can be processed into fibrous forms for reinforcing plastics.

SIZE EVALUATIONS

Evaluation of a size consists in surface-treating the fibers, processing the fibers stepwise through the requisite operations, fabricating a reinforced plastic part and measuring the properties of the part.

Evaluation of the handling characteristics of glass fibers is an art. The processing must be carried out on standard, production type equipment. The evaluation of the handling of the fibers through each process operation must be done by an operator who is experienced in the process, knows what to look for, is on the lookout for unexpected behavior and can judge how well the fibers are handling.

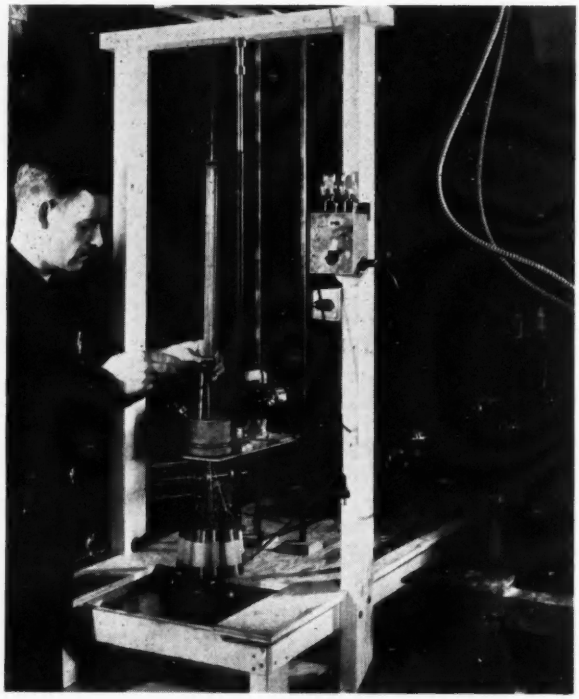
Evaluation of the performance characteristics imparted to the glass-plastic part by the sized fibers is carried out in a preliminary manner by reinforcing plastic

rods with parallel strands, which have been surface-treated during forming of the fibers. This evaluation of sizes has been accomplished in the past by forming strands, cutting the strands and preforming mats. These mats were used to reinforce plastic panels. It was found that, in order to obtain meaningful data, many preforms and panels had to be fabricated and tested. The variation in physical properties found for panels molded in one press under identical conditions is primarily due to the variation in orientation and concentration of strands in the individual test specimens. The reinforced-plastic-rod method of testing was adopted to eliminate these and other variables inherent in the fabrication and testing of preformed, flat glass-plastic panels. Experience obtained in the fabricating and testing of a large number of preformed panels and rods has shown that the rod technique is the better and faster method for preliminary evaluation of the performance characteristics of glass fibers.

For evaluation of a size, a strand from a dried forming-package is wound on a small creel. For evaluation of a finish, a strand is wound on the creel and given the desired surface treatment. The strand on the creel is then impregnated with an acetone solution of a diallylphthalate-type polyester plastic. The concentration of the plastic solution is controlled to give the desired glass-to-resin ratio in the finished rod. The impregnated strand is air-dried for 30 minutes followed by 45 minutes in an oven at 130°F to remove all the acetone. If a styrene-polyester type plastic is used, modifications in strand impregnating technique are necessary. Also, control of styrene loss due to evaporation during rod fabrication is required.

The apparatus for fabricating test rods is shown in Figure 5. The impregnated strand is strung up over a hook in loops. A weight is hung at the bottom of each loop to put the strand-loops under equal tension. A motor-driven wrapping device winds ¼-inch wide cellophane tape under constant tension around the suspended strand-loops. While the strand-loops are still under tension, a portion of the fashioned rod is then clamped in a special jig. The jig and rod are placed in a curing oven at 180°F for 30 minutes and then at 230°F for 60 minutes.

The composition of the rod is measured by loss on ignition at 1000°F. The water absorption is measured by increase in weight after immersion in water for 7 days. Ultimate flexural strength is measured for the dry and wet rods after immersion in water for 7 days or after boiling for 2 hours. Modulus in flexure is obtained for the flexural strengths measured.



(Owens-Corning Fiberglas Corp)

Figure 5
Test Rod Fabrication
Surface Treatments for Fibrous Glass

These rod properties give an excellent preliminary indication of the relative performance characteristics of the fibers for use in reinforcing polyester plastics. Fibers which impart high strength to diallylphthalate type polyester plastic rods also give high strength to styrene-polyester type rods.

Fibers which have good handling characteristics and show up well in the rod tests are evaluated further for performance characteristics. A considerable number of preform moldings are made and tested. Cut-strand-mat and parallel-strand-mat are formed and fabricated into reinforced polyester plastic parts and tested. If the fibers show up well in all these evaluation tests, then the fibers in various forms are submitted to customers for field evaluation. If field tests are satisfactory, the fibers are recommended for production and sale as new and improved fibrous glass products for reinforcing polyester plastics.

SIZE COMPARISON

Variation in size composition causes variation in the handling and performance characteristics of fibers. This is illustrated by comparison of characteristics of fibers manufactured with four different sizes, A, B, C and D.

"A" is a good textile size. The fibers handle well through twisting, plying and weaving; they cannot be used to reinforce polyesters unless they are given a suitable finish. "B" is a good polyester plastic reinforcing size; the fibers handle poorly through twisting, plying and weaving. "C" and "D" are good polyester plastic reinforcing sizes. The fibers sized with "C" handle fairly well through twisting, plying and weaving; however, the handling characteristics in these operations are not good enough for economical processing. Fibers sized with "D" are poor in twisting, plying and weaving.

Size "A" is composed of many ingredients blended together in a water medium to give a surface treatment to the fibers so that they can be used in all the common textile processing operations. The principal ingredients are starch, vegetable oil, softener, gelatin, vinyl polymer and emulsifying agent. The starch, gelatin and vinyl polymer are the film-forming adhesive, bulking and surface protectant agents for the fibers. The vegetable oil and softener act as softener and lubricant for the fibers. The emulsifying agent is used to disperse the oil in the water.

The principal constituents of size "B" are a plasticized vinyl polymer, a glass lubricant and an organo-metallic complex compound dispersed in water. The plasticized vinyl polymer, which is different from the one used in size "A", performs the same function on the fibers as do the starch, gelatin and vinyl poly-

TABLE I
HANDLING CHARACTERISTICS
Fibrous Glass Surface-treated During Forming

Surface Treatment	Forming	Twisting	Winding	Weaving	Creeling	Roving	Cutting	Preforming
Size A	Good	Good	Good	Good	Poor	Poor	Poor	Poor
Size B	Good	Poor	Poor	Poor	Good	Good	Good	Good
Size C	Good	Fair	Fair	Fair	Good	Good	Fair	Fair
Size D	Good	Poor	Poor	Poor	Good	Good	Fair	Fair

mer in size "A". The lubricant is used in place of the oil and softener in size "A". Its lubricating effect is less powerful than that obtained with the oil and softener. The organo-metallic complex acts as the coupling agent to bond the fibrous glass surface to the polyester plastic.

Size "C" contains an elastomer, a glass lubricant and an organo-silicon polymer dispersed in water. The stability of this size is better than the stability of the other three. The elastomer serves the same purpose as the plasticized vinyl polymer in size "B". It forms a film which is more water resistant than that formed by size "A" or size "B". The lubricant is less effective than the lubricant in size "A". The organo-silicon polymer is the coupling agent, giving better bonding between the fiber surface and the polyester than the coupling agent in size "B"; the bonds are very resistant to the action of water.

The lubricant in size "D" is similar to that in size "B". A plasticized vinyl polymer is used in "D" which forms a more moisture resistant film than the polymer used in "B". The film formed by this polymer does not discolor as readily by heat as does the elastomeric film. The coupling agent in "D" is the same as in "C". The handling and performance characteristics of fibers sized with "C" are very similar to those sized with "D".

Table I shows a qualitative comparison of the handling characteristics of fibers which have been treated with the four sizes. The behaviors of these fibers in the processing operations are classed as "poor", "fair" and "good". Plying is omitted because it is a second twisting operation. The cut-strand-mat process is omitted because behavior of fibers in this operation is similar to preforming. The parallel-strand-mat process is omitted because handling characteristics of fibers in creeling and roving cover handling characteristics in this operation.

Fibers which are surface-treated with size "A" form a strand which is pliable, well lubricated, abrasion resistant, and has fair wet strength. The fibers handle well in twisting, plying and weaving. However, they handle poorly in roving, cutting, preforming and cut-strand-mat formation; the fibers are too well lubricated for these operations. The strands open up during the cutting and preforming operations because the filament-to-filament adhesion is poor. This is due to a combination of the weak film and oil on the fiber surface. Fibers which are surface-treated with size "B" handle poorly through the operations required to fabricate yarns and woven fabrics; the abrasion resistance and lubrication of these fibers are too low. The low level of lubrication combined with the right degree of filament bonding and pliability enables the fibers to process well through the other operations required to convert fibers into forms useful for reinforcing polyesters.

Fibers formed with size "C" are only fair in twisting, plying, weaving, cutting and preforming. They process well in forming, creeling and roving. The lubricant present in the size is not quite good enough to allow good yarn and cloth fabrication and a little too efficient to permit good cutting and preforming operations. These fibers wet out more completely but not as rapidly as fibers treated with size "B".

Fibers sized with "D" handle like those sized with "B" in twisting, plying and weaving. This is because the film-forming and lubricating constituents are similar. Fibers sized with "D" do not cut nor preform as well as those sized with "B"; the film formed by size "D" does not have the right degree of flexibility. These fibers have wetting properties similar to those sized with "B".

Some of the performance characteristics of fibers which are surface-treated with the four sizes are shown in Table II. The glass content for all rods was comparable. Results obtained with fibers given various finishes are shown for comparison. Results obtained by finishing with a mixture of vinyl trichlorosilane and beta chlorallyl alcohol, as recommended by the Bjorksten Research Laboratories, are given for comparison. This finish is designated as "Modified VTS".

The data were obtained by the procedure described in the discussion of Size Evaluation. Examination of these data shows that, for polyester reinforcement, fibers surface-treated during forming with sizes "C" and "D" are superior to those sized with "A" and "B". Finish "136" is one of the best surface treatments to apply to yarns and fabrics. Results obtained with this finish are comparable to results obtained with the best sizes for polyester-reinforcing glass fibers.

Fibers sized with "A" give the polyester rods low ultimate flexural strengths and moduli. This is due to poor glass-to-plastic adhesion caused by (a) the absence of a coupling agent, (b) the presence of too much lubricant, and (c) the presence of an incompatible film on the fiber surface.

Fibers sized with "B" give better dry strengths to the rods than those sized with "A"; however, the water absorption is high and the wet flexural strength and modulus are low. The film formed by size "B" has low moisture resistance and the bonds formed by the coupling agent are weakened by water.

Fibers sized with "C" and "D" have nearly equivalent performance characteristics. The dry and wet properties of polyester rods reinforced with these fibers are much better than those reinforced with fibers sized with "A" and "B". The water absorption by these rods is much lower. The good performance characteristics shown by these fibers are the result of a surface coating containing (a) a good coupling agent (b) a compatible, water-resistant film, and (c) a weak lubricant.

CONCLUSION

Good surface treatments for fibrous

TABLE II
PERFORMANCE CHARACTERISTICS
Polyester Rods Reinforced With Surface-treated Fibrous Glass

Surface Treatment	Dry Strength Flex 10^3 psi	Dry Strength Flex Mod 10^6 psi	Wet Strength Flex 10^3 psi	Wet Strength Flex Mod 10^6 psi	Wet Strength Retention %	Water Abs Wt %	Plastic Wt %
Size A	80	5	62	4	78	1.0	40
Size B	166	6	54	4	33	1.1	34
Size C	218	7	206	7	95	0.4	28
Size D	202	6	191	6	94	0.4	31
Finish 111*	153	8	107	5	70	0.9	38
Finish 112*	175	7	36	1	21	0.6	28
Finish 113*	206	9	148	6	72	0.8	33
Finish 114*	182	7	141	6	78	0.3	27
Finish 136*	247	8	222	7	92	0.3	29
Modified VTS	239	7	213	7	89	0.2	32

* Finish designations of Owens-Corning Fiberglas Corporation

glass that will be used for reinforcing polyester plastics have been developed. One kind of surface treatment applied to fibers as they are formed is necessary for satisfactory fabrication of good yarns and woven fabrics. Another kind of surface treatment of the yarns and fabrics is required before dependable reinforcement of polyester plastics is obtained.

Surface treatments have been found which are applied to the fibers as they are formed and enable satisfactory fabrication of fibers into forms suitable for reinforcing plastics without additional surface treatment.

The "universal size" has not been discovered, but its properties are known. The kind of size suitable for fibers to be twisted, plyed and woven is different from the kind of size suitable for fibers for reinforcing plastics. The type of size that is good for fibers used to reinforce one type of plastic is not satisfactory for fibers used to reinforce another type.

The development of new surface treatments for fibrous glass during the past year has made possible the fabrication of glass-reinforced polyester plastics with improved properties. Future research on surface treatments for fibrous glass should result in further improvement in the properties of reinforced polyester plastics. New surface treatments designed for fibers suitable for reinforcing other types of plastics will be developed.

• PATENT DIGEST •

PAUL WENGRAF

WATER REPELLENT FABRICS——UREA-GLYOXAL—Formaldehyde Condensates Condensed with Higher Fatty Alcohols G, 2, 02

U S Pat 2,588,640
(Bozel Maletra——Lehmann, Lintner——March 11, 1952)

Co-pending U S Pat 2,574,114 (cf *Am Dyestuff Reptr 41,* 306 (1952)) protects a shrink-proofing and stabilizing treatment, which consists of applying condensates of urea that are reacted with glyoxal until a portion (about 50%) of glyoxal is fixed and thereupon condensing the product with formaldehyde in alkaline solutions. Durable methylol-type products are obtained which are used for impregnating textiles in the presence of ammonium nitrate as a catalyst.

The present modification of this invention incorporates higher fatty alcohols into emulsions of the resin-type condensates. A small amount of emulsifying agent (e g, a fatty alcohol sulfate) may be dissolved in an aqueous solution of the condensate and then emulsified with a higher fatty alcohol of a 15 to 20 Carbon chain length (e g, octadecylalcohol) at such a ratio that only part of the methylol groups undergo etherification, which is effected by acidifying and refluxing the emulsion. It is suggested that to improve the stability of these emulsions, a nonionic assistant, such as the condensation product of one mol octadecanol with about 20 mols ethylene oxide (Emulphor), be added.

Example: Glyoxal diurea is prepared by dissolving 1.6 kg urea in 5.8 kg of a dilute glyoxal solution and heating in the presence of some HCl. The white precipitate is further heated with alkaline formaldehyde. First, a small amount of octadecyl sulfate of sodium is added to this emulsion while it is being stirred at 70-80° C, and thereafter n-octadecanol is added. The composition is acidified with acetic acid and refluxed. Later some octadecanol-ethylene oxide condensate and a small quantity of paraffin (m p 43-46° C) are added. The homogenized mixture is stirred in cold water to give a total concentration of 5% of solids. Fabrics prepared with this emulsion, dried at 90° C and baked at about 120° C become durably water-repellent. The effect is said to persist even after five washings in boiling soap-soda ash solution. Moreover the soft handle of the original material reportedly remains unchanged.

References cited by the Patent Office:

U S Pat 2,427,504 (American Maize Products/1947): aldehyde-reacted prolamine base proteins, such as zein, gliadine, and the like are mixed with asymmetrically-substituted ureas, such as N-N-diethanol urea, to control the otherwise irregular reaction.

Brit P 494,700 Du Pont/1938): urea is heated with a saturated, nonhydroxylated aliphatic aldehyde other than formaldehyde at a temperature above 50° C in the absence of any catalyst and in the presence of a monohydric alcohol (e g, urea-butanol-propionaldehyde) to obtain hard colorless films.

SHRINKPROOFING WOOLEN GARMENTS——Dry Chlorination under Controlled Moisture Conditions A, 2, 04

U S Pat 2,590,811
(Forstman Woolen Co——Zimmerman, et al ——March 25, 1952)

The preamble to this specification quotes sources pertaining to a series of wool chlorination processes assigned to Wool Ind Res Ass'n, such as Brit Patents 417,719 and 475,742, which recommended that the treatment be carried out in an evacuated chamber; Brit P 493,098, which advocates aftertreating with sulfur dioxide as the acidifying agent; and U S Pat 2,373,974 which refers to the chlorination of wool containing less than 9% moisture and a papain solution buffered to pH 5. Many other pertinent processes are also cited in the Patent Office's references.

Thus, during the application of former processes of this type, the opinion prevailed that the moisture content of woolen goods that were to be subjected to dry chlorination must not exceed 9% because of the risk of irregular results and the development of free HC1 with damage to the wool material and to the cotton seams used for holding the woolen parts of the garments together.

Surprisingly it has been observed that shrinkproofing of woolen garments, such as knit sweaters or underwear may be greatly improved by the use of goods with a moisture content of 10.5—12%, provided that irregular condensation of HC1 on the goods is prevented. Chlorinating may be carried out in an air-tight, pressure-resistant chamber, which preferably is evacuated before chlorine gas is admitted. The garments are placed on hangers in this chamber. According to the examples cited, a lot of sweaters having respective moisture contents of 10.5%, 11% and 11.75% shrunk 8%, 4.3% and 1.4% in length after being washed in hot water.

DYEING WITH CONCENTRATED VAT ACID PASTES C, 4, 02

U S Pat 2,590,213
(Sandor——March 25, 1952)

The preamble to this patent discusses various difficulties encountered in normal

vat dyeing procedure, mainly, poor stability of the dyebath, insufficient dispersion (e g, in pigment pad dyeing), lack of substantivity of Indigosol dyes, etc. Most of these are reportedly overcome through use of the vat acid process. The inventor states, however, that the prepartion of these colloidally-dispersed dyebath solutions requires not only thorough knowledge of the properties of the individual dye brands but also permanent control of the process. Moreover, the depth of shade is said to be limited and combinations of two or more dyes present further problems.

According to this patent, a concentrated, stable vat acid is obtained by mixing the paste or powder of a vat dye with a relatively small amount of water (e g, one p dyestuff to 5 p water) and adding a reducing agent. In this step the dye is reduced but not yet dissolved; a protective agent, such as sodium lignin sulfonate, a quaternary ammonium compound and a fatty alcohol sulfate are added following neutralization with acid, and water is evaporated under reduced pressure at temperatures not exceeding 60-70° C. Pastes of this type can be used in padding processes at relatively high concentrations: for example, by padding with a paste containing 16% of vat acid, dispersed 188: 1000 in water, goods squeezed to a pick up of 100% will contain 30 g dye per liter, i e, 3%. Up to now it has not been possible to obtain shades of this intensity in the vat acid dyeing process.

Example: a vat dye, e g, Indanthrene Blue RS or Pyranthrone, are vatted according to standard procedure with alkali and hydrosulfite in the presence of sodium lignin sulfonate. The water content is not sufficient to dissolve the leuco compound formed. Alkali is neutralized with formic acid, the resulting collodial solution is filtered and concentrated in vacuo to a paste containing 16% solid dyestuff. A fabric padded in a 0.6% dispersion of this paste is dried in the hot flue, prepared with NaOH, hydrosulfite and common salt, and steamed. Jig dyeing is said to be less suitable because of the difficulty of maintaining exactly the same dyeing conditions for each batch.

The process might also be used in printing, wherein the goods are printed with the vat acid, thickened with methyl cellulose, for example, dried, padded in hydrosulfite and aged as usual.

Among other references cited by the Patent Office:

U S Pat 2,227,834 (General Aniline & Film/1941): mixtures of benzanthone vat dyes with yellow anthraquinoid dyestuffs (U S Pat 1,459,536) become fast to light without fading of the yellow component.

U S Pat 2,193,751 (Ciba/1940): valuable gray to black prints are obtained by printing mixtures of blue to violet benzanthrone dyes with indigoid dyes, e g, naptho-thioindigo products.

U S Pat 2,107,526 (Dreyfus): padding a cellulose acetate fabric with a neutral or acid leuco-compound of a vat dye, partially drying in an oxygen-free atmosphere and reoxidizing. Preparation of the leuco compounds is described in U S Pat 1,900,-172 (Celanese/1933), which advocates coloring cellulose ester fabrics with free leuco compounds, preferably in dispersed state. These compositions are prepared by pasting a vat dye with a dispersant, reducing in alkaline solution, adding the dispersion to the dyebath and carefully acidifying.

Br P 358,439 (I G Farbenindustrie): dyestuffs of the anthraquinoid or thioindigoid series may be converted into stable leuco preparations by mixing the leuco compound with molasses or other protective colloids.

SHRINKPROOFING WOOL—— Chloramides and Chloro-Sufonamides Used A, 2, 04

U S Pat 2,590,390
(Wolsey Ltd——Earland, et al——March 25, 1952)

Solutions of chlorine or bromine as well as those of hypohalous acids are known to act rapidly on wool even at room temperature, thus causing uneven treatments. The inventors in this patent mention other processes, e g, that of Brit P 595,518 (Alrose Chem Co), which describes a chlorinating method using alkyl sulfonchloramide plus formaldehyde, chloramines or bromamines and other halogenated compounds. (The British Patent is apparently parallel to USP 2,427,097 of Kamlet et al (see *Am Dyestuff Reptr* 36, 788 (1947), which mentions, among others, halogenated sulfonamides, such as p-toluene sulfonamides and similar products). Chloramines are said to have an insufficient shrinkproofing effect. Moreover they are known to produce yellowing of the wool.

It has been found that the best compounds for rendering wool shrinkproof in a controlled manner are chloramides, chlorosulfonamides, dichloramides or dichlorosulfonamides, which do not hydrolyze to sulfonamines. For example N:N'-dichlorourea, tending to hydrolyze to chloramines, is not satisfactory in contrast to N-dichloro toluene sulfonamide . . . CH_3-$C_6H_4SO_2NCl_2$ . . . or N-N' dichloro-biuret . . . NH: $(CO.NHCl)_2$. These products may be prepared and isolated or they may be obtained in situ by mixing the corresponding amide with hypochloric acid:

$R.CONH_2 + HOCl \longrightarrow RCONHCl + H_2O$.

2½-4% available chlorine (calculated from the amount of wool) are generally sufficient to make wool unshrinkable, but the aforementioned chlorinated amides need about twice as much chlorine, e g, an amount of monochlorurea corresponding to 6-8% Cl is required. It has been discovered, however, that the percentage of available chlorine can be reduced (to 2-4%) by adding chloride ions in the form of soluble chlorides, preferably NaCl. The pH of the bath must not exceed 2.0 and the chloride ion concentration has to be kept above 0.3 N. Under these conditions the rate of reaction is five times slower than is the case when a solution of free chlorine or bromine is applied. By reason of this slower reaction the procedure permits working at elevated temperatures (about 40° C) with even penetration and good results. Tables show the effect of various acids with and without the addition of chlorides.

Among other references cited by the Patent Office:

U S Pat 2,429,082 (Stevenson Dyers/1947—cf *Am Dyestuff Reptr* 37, 383 (1948); a shrinkproofing treatment of wool consisting of a chlorine liberating agent and permanganate.

U S Pat 2,414,704 (Du Pont/ 1947—cf *Am Dyestuff Reptr* 36, 419, (1947)): reducing the shrinking tendency of wool by immersing the goods in an aqueous solution of 1-3-dichloro-5-5'-dimethyl hydantoin and adjusting to a pH of 1-3.

U S Pat 2,144,824 (Chem Fabrik Grunau/1939): chlorinating wool with hypochlorite in the presence of ammonia, amines or nitrogenous-fiber protecting agents, such as degradation products of proteins (the so-called "lysalbinates").

U S Pat 1,892,548 (Feibelman/1932): bleaching with p-toluene sulfondichloroamide or similar products ("Aktivin").

Brit P 549,362 (Imperial Chemical Industries/1942): mothproofing wool with an acidified aqueous solution of formaldehyde at a pH not higher than 1.0, washing free of formaldehyde and acid and drying.

DYEING WOOL WITH HIGH AFFINITY COLLOIDAL DYES——— Dimmonium Phosphate Added C, 4, 06

U S Pat 2,590,847
(Pacific Mills——Dalton, Ploubides——Apr 1, 1952)

"High-affinity collodial dyes" are defined as wash-fast wool dyestuff types having excellent affinity for the fiber and acting very rapidly but tending by the same reason to produce uneven results. The dyes applied according to the present process comprise chromatable dyes, e g, Alizarine Brown BL or Omega Chrome Orange ML, metachrome dyes or milling

dyes, such as Polar Red G, Polar Orange R, Alizarine Milling BL, etc.

Woolen fabrics, yarns, slivers or raw wool fibers are entered into an aqueous dyebath made distinctly alkaline at the start by the addition of diammonium phosphate; this bath remains alkaline for a substantial part of the process. The alkaline condition causes retention of the dye in fine dispersion and prevents fixation on the fiber. In this stage the wool is slightly swollen, whereby penetration is promoted. After about one to 1½ hours heating below the boiling point the temperature is raised to the boil; gradually ammonia evolves and the bath becomes more and more acid. (A table reports on the pH values measured during the whole dyeing operation). Initially the pH is at 7.85, dropping for the first hour (at 185° F) to 7.30 and then by continued boiling to 5.78.

Principally, the same procedure is used whether the wool has to be pre-chromed, afterchromed or dyed according to the metachrome process or with one of the milling dyestuffs. A modification of this method (described in one of the examples) consists of increasing the acidity during boiling after a certain time by adding ammonium sulfate to the diammonium phosphate-containing dyebath.

Among other references cited by the Patent Office:

Brit P 588,454 (Noble/1947): textiles containing wool are dyed with acid, chrome- or metalized dyes by adding o-phosphoric acid (instead of other acids) or a salt thereof. Goodall, in his article "Theory and Practice in Wool Dyeing" (*Am Dyestuff Reptr 36,* 384 (1947)) suggests alterations of standard dyeing procedure for Eriochrome or Polar dyes such as entering the goods as near the boil as possible, omitting Glauber's salt and reducing the acid content. This article also compares absorptions of dyes in acid baths containing sulfuric, hydrochloric or phosphoric acids.

Noble, in his article "pH Control in Dyeing Processes" (*Am Dyestuff Reptr 34,* 359, 367 (1945) discusses the buffering action of phosphoric acid and phosphates for controlling pH values in pretreatments and dyeings; chrome- and acid colors are advantageously dyed with mixtures of phosphoric acid and ammonia, preferably in the ratio of monoammonium phosphate.

Further reference is made to Bucherer's German P 591,212, which recommends that chrome dyestuffs be dyed on wool in an ammoniacal solution containing some ammonium carbonate; diammonium phosphate is not mentioned in this patent.

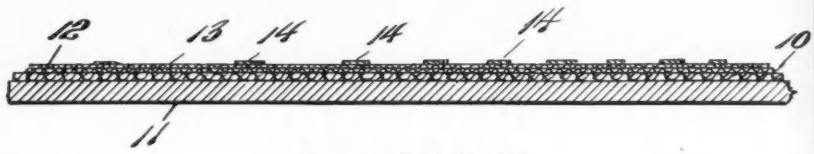


Figure 1—USP 2,590,643

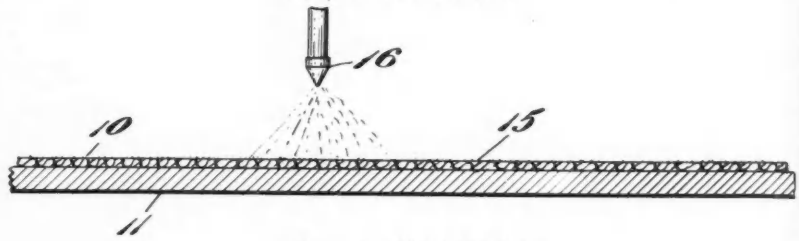


Figure 2—USP 2,590,643

SCREEN PRINTING BACK GREIGE BLANKET D, 4

U S Pat 2,590,643
(Owren, Koralczuk——March 25, 1952)

Figure 1 of the accompanying drawing shows the usual elements used in screen printing: the back greige or gray (10) pinned on table (11), the cloth to be printed (12) and the screen (13) placed upon the cloth (12). It is frequently claimed that the backing cloth ("back-greige") unevenly absorbs the color penetrating the printed fabrics, thereby causing printing faults such as stick marks. Moreover, the back cloth has to be washed regularly between changes of colors.

Figure 2 illustrates the present modification. The contacting surface of the back greige (15) is sprayed by means of nozzle (16) with a dye repellent substance. Usually, aqueous solutions or dispersions of the dyestuffs are employed; in this event water repellent coatings, such as aluminum stearate, are used for covering the surface. When oil soluble dye preparations are used, the coating composition accordingly has to be replaced by an oil-resistant layer. The filaments of the nap of surface (15) must be coated with repellent while the interstices between the threads of the back greige must remain open. It is suggested that the surface be coated prior to any color change with a new protecting layer of the repellent composition in order to avoid repeated washing and to prevent premature attrition of the back cloth.

References cited by the Patent Office:

U S Pat 1,231,141 (Ellis/1917): a printer's blanket composed of a layer of felt or of felt-like fabrics pasted on a thin woven fabric by an oil-proof cement. The outside of the thin fabric is covered with a stain-resistant lacquer coating.

U S Pat 1,210,375 (Tingue, Brown & Co/1916): a printer's blanket comprising a felt or felt-like fabric covered with an oil-repellent material, particularly a metal sprayed in the form of fine particles against one side of the felt which has previously been impregnated with a suitable adhesive.

LUSTROUS PRINTED CALENDER EFFECTS——— Vat Acid Compositions D, 2, 01

U S Pat 2,592,852
(Jos Bancroft & Sons——Billingsley, Jr, Hall ——Apr 15, 1952)

Pigment-printed patterns are known to mark off when passing through a mechanical finishing device. This drawback becomes even more serious when a frictionating calender operating at relatively high temperatures is used in order to produce a glazing effect. The present invention introduces a method by which vat dyes may be printed on fabrics without the risk of smearing or marking-off in a subsequent strong calendering process.

This problem has been solved by incorporating vat acid compositions into a printing paste. The vat acids are produced according to the standard method of reducing the vat dye in alkaline solutions in the presence of a dispersing agent, e g, "Blancol" (sodium naphthalene sulfonate formaldehyde), then carefully neutralizing with formic or acetic acid to the neutral or slightly acid state, and mixing this dispersion with a thickening agent and a resin-forming compound, such as "Aerotex M-3" (methylated methylolmelamine), a potentially acid compound, such as ammonium thiocyanate, as a curing promoting catalyst, and a lubricant (Carbowax 1500) to prevent sticking of the cloth between the heated rollers. After the printing and drying operations, the goods are optionally cooled and passed several times between heated rollers of a regular calender or frictionated. The lustrous effect extends over the entire surface of the

(Concluded on Page 525)

Proceedings of the

American Association of Textile Chemists and Colorists

Copyright, 1952, American Association of Textile Chemists and Colorists

Editor of Proceedings: Carl Z Draves, 30 Lahey St, New Hyde Park, N Y

August 18, 1952

CALENDAR

COUNCIL
September 12 (Philadelphia); November 6 (Boston), Jan 16, 1953; April 17, 1953; June 5, 1953.

GENERAL RESEARCH COMMITTEE
(See Council dates)

NATIONAL CONVENTIONS
November 6-8, 1952 (Hotel Statler, Boston); September 17-19, 1953 (Hotel Conrad Hilton, Chicago); 1954 (Atlanta, Ga.); Sept 21-25, 1955 (Chalfonte-Haddon Hall, Atlantic City, N J).

MID-WEST SECTION
October 4 (Hotel Bismarck, Chicago).

NEW YORK SECTION
September 26, October 24, November 21 (Kohler's Swiss Chalet, Rochelle Park, N J); January 30, February 27 (Hotel Statler, New York, N Y); April 17, May 22 (Swiss Chalet)

NORTHERN NEW ENGLAND SECTION
December 6 (Annual Meeting, Lowell Textile Institute).

PACIFIC SOUTHWEST SECTION
October 4 (Outing, Ojai, California).

PHILADELPHIA SECTION
Sept 12, Oct 24 (Penn Sheraton Hotel); Dec 5, Jan 16 (Kugler's Restaurant).

PIEDMONT SECTION
Sept 13 (Hotel Charlotte, Charlotte, N C).

RHODE ISLAND SECTION
Oct 24 (Providence Engineering Society); December 5 (Annual Meeting, Sheraton-Biltmore, Providence, R I).

SOUTH CENTRAL SECTION
Aug 22-23 (Outing, Chattanooga Golf & Country Club); December 6 (Patten Hotel).

SOUTHEASTERN SECTION
Sept 13 (Biltmore Hotel, Atlanta).

WESTERN NEW ENGLAND SECTION
Oct 3, Nov 14 and Dec 12 (Rapp's Restaurant, Shelton, Conn).

Addendum

AT the time of publication of the recent 172nd General Research Committee meeting, the report of the Subcommittee on bleaching was unavailable. The report follows:

SUBCOMMITTEE ON BLEACHING—T E Bell, Chairman, reports that his committee is actively at work. Samples of bleached cotton goods are now undergoing physical tests. Fluidity data are also being gathered. A study is in progress on the use of the spectrophotometer as a method of measuring whiteness. It is hoped at the September meeting to review all data and consider the possibility of submitting tentative standards.

EMPLOYMENT REGISTER

This column is open for two insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the Secretary of the Association, Lowell Textile Institute, Lowell, Mass. It is understood that these will be open to inspection by prospective employers who can obtain further information from the Secretary.

52-16
Education: B S, Bradford Durfee Tech Inst, Chemistry & Dyeing; now graduate student, Polytechnic Institute of Brooklyn.
Experience: not technical, references.
Age: 27; single; work as textile chemist in vicinity of Brooklyn desired.
8-18

52-17
Education: Lowell Evening Textile School and university extension.
Experience: assistant dyer, wool.
Age: 25; single; references; northeast U S preferred, but not essential.
8-18, 9-1

52-18
Education, College, (evening), chemistry.
Experience: chemist, laboratory head, plant manager.
Age: 30; married, references; New York or northeast U S preferred.
8-18, 9-1

OTHER EVENTS

AMERICAN CHEMICAL SOCIETY
7th National Chemical Exposition, Sept 9-13, Chicago Coliseum, Chicago, Ill. This will include the 2nd International Art Exhibit.

AMERICAN SOCIETY FOR TESTING MATERIALS
1953 Annual Meeting, June 29-July 3, Chalfonte-Haddon Hall, Atlantic City, N J.

AMERICAN STANDARDS ASSOCIATION
3rd National Standardization Conference, Sept 8-10, Museum of Science and Industry, Chicago, Ill.
34th Annual Meeting, November 19, Waldorf-Astoria, New York, N Y.

AMERICAN TEXTILE MACHINERY ASSOCIATION
Exhibit of American Textile Machinery, April 26-May 1, 1954, Atlantic City Exhibition Hall, Atlantic City, N J.

CANADIAN TEXTILE SEMINAR
September 11-13, Queen's University, Kingston, Ont.

FIBER SOCIETY
Sept 10-11 (Princeton, N J).

INTERNATIONAL COTTON AND RAYON EXHIBITION
Sept 27-Oct 12, Busto Arsizio, Italy.

LEIPZIG FAIR
September 7-17, Leipzig, Germany.

NATIONAL ASSOCIATION OF HOSIERY MANUFACTURERS
41st Knitting Arts Exhibition, April 27-May 1, 1953, Atlantic City Auditorium, Atlantic City, N J.

NEW YORK BOARD OF TRADE (DCAT)
26th Annual Meeting: September 25-28, Pocono Manor Inn, Pocono Manor, Pa.
27th Annual Drug, Chemical and Allied Trades Dinner, March 5 (1953), Waldorf-Astoria, New York, N Y.

TEXTILE RESEARCH INSTITUTE
23rd Annual Meeting, Nov. 17-18, Hotel Commodore, New York, N. Y.

Hudson-Mohawk Section——

DYEING ORLON*

E SZLOSBERG
Sandoz Chemical Works, Inc

INTRODUCTION

SINCE the first application of cuprous-ion technique to the dyeing of Orlon acrylic fiber, the tentative procedures have been tested in a number of practical mill runs. These various demonstrations on both small and large lots have given us opportunity to introduce certain advantageous changes in the procedures, a number of which will be discussed in this paper.

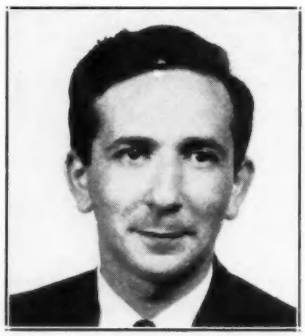
It is known that Orlon stock, yarns and fabrics can be dyed with comparative ease when reduced copper is introduced into the bath. The copper dyeing method, which was first applied to dynel, is today a well-established solution to the problem of dyeing Orlon acrylic fiber. It is true, however, as originally stated by Carbide & Carbon Chemical Company, that the effectiveness of this method decreases as the proportion of acrylonitrile to other copolymer increases. Thus, dynel, which contains only 40% of acrylonitrile in its molecule, lends itself more easily to copper dyeing than Orlon, which is a polymer of acrylonitrile alone.

EXPERIMENTAL

There are at present two types of Orlon acrylic fiber produced by DuPont:

a) Orlon 81—all-filament fiber
b) Orlon 41—staple fiber

The dyeing properties of these two types of Orlon acrylic fibers differ to a large extent, and the results obtained on 41-type Orlon could not be reproduced on the 81 type. Comparative dyeings of Orlon 81 and 41 (Figure 1) show that different types of Orlon exhibit different absorption rates relative to acrylonitrile content and degree of orientation. It must be stressed here that the difference in absorption rates between 81- and 41-type Orlon is of a decisive value, and, while the latter produces fast-to-washing shades (including navies, maroons, dark greens and blacks) with reduced copper in the bath, 81-type filament Orlon gives a much reduced exhaustion and wet fastness. At its present stage of development the dyeing of Orlon depends upon adding to the dyebath a reduced copper salt, which is absorbed by the fiber at a rate dependent upon several factors. The rate and the amount of copper absorption will obviously determine the ease or difficulty with which a selected dye or dyes are absorbed.



E Szlosberg

Mill runs and laboratory experiments have permitted improvements to be made in the tentative procedures for dyeing Orlon acrylic fiber. Control of pH as well as temperature is important in the application of acid dyes on Orlon, and dyes in mixtures must behave similarly with respect to saturation of the fiber to get consistent results. Hydroxylammonium sulfate is preferred as a reducing agent for the preparation of cuprous ions, which must be present in the dyebath for dyeing Orlon satisfactorily. The drip method of addition of cupric salt and reducing agent has been found desirable for attainment of better uniformity and exhaustion in dyeing, especially for large lots.

The rate of copper absorption by Orlon depends mainly on the following factors:

a) pH value of the bath
b) Temperature
c) Time

The pH value of the dyebath is of decisive importance in dyeing Orlon acrylic fiber and thus must be controlled throughout the dyeing operation. There is only a limited range of pH values at which a successful dyeing can be made. When pH value of dyebath is plotted against absorption of copper by Orlon acrylic fiber, it is found that the absorption of copper gradually increases as the pH value rises from 2 to 4, becoming approximately constant at pH 4. When the pH of the bath drops below 2, the amount of copper absorption diminishes considerably, and, in consequence, only surface dyeing takes place. Theoretically, then, the optimum range of pH values is within the limits of 2 to 4. In practice, however, it is found that the reduction of cupric sulfate by hydroxylammonium sulfate proceeds too rapidly if the pH value is allowed to rise above 3, and thus precipitation may occur. The practical methods of keeping pH values within this range will be indicated later.

Figure 2 shows comparative dyeings of Orlon 81 and 41 made at 180°F, 200°F and 212°F. It is evident that the amount of dye absorbed by the fiber (and therefore the amount of copper absorbed) is proportional to the temperature of dyeing. In the case of Orlon 81 the bulk of dyeing takes place at temperatures close to 212°F, and thus actual boiling temperatures are necessary for successful operations.

Time element plays an important part in exhaustion of color. Since the reduction of cupric sulfate to its cuprous form proceeds gradually and the fixation of color on the fiber follows with a time lag, the time required for best exhaustion must necessarily be prolonged.

AFFINITY OF DYES———Among the various classes of dyes that can be applied to Orlon fiber under various conditions of temperature the following are selected:

colors	
basic	chrome
acetate	direct
acid	vat

In this classification the most satisfactory results have been obtained with selected acid colors. In this group there are many dyes with good affinity for Orlon 41, but a limited number producing full shades on Orlon 81. For the latter, at present, only several dyestuffs can be found with satisfactory fastness properties. Among them are the following:

Alizarine Light Blue 2A	Pr 12
Rocceline S	CI 176
Orange Y	CI 151
Chinoline Yellow O Conc	CI 801
Xylene Milling Yellow S	CI 145

* Presented before the Hudson-Mohawk Section in Albany, N Y, on May 2, 1952.

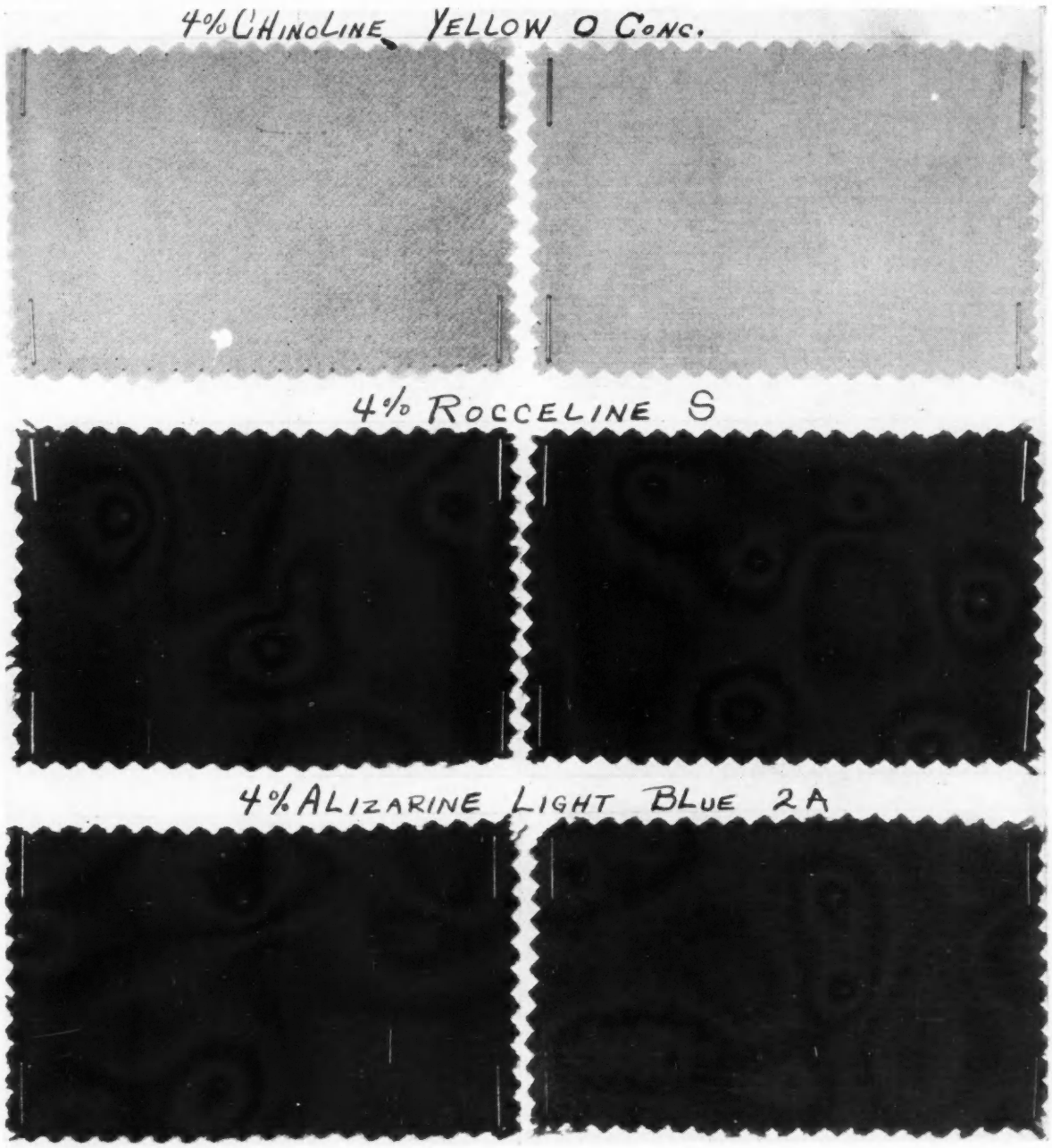


Figure 1
Strength Comparisons
Orlon 41 (left) versus Orlon 81 (right)
Dyed by Drip Method
4% dye
10% cupric sulfate
4% hydroxylammonium

A large number of acid colors were tested on 41-type Orlon acrylic fiber, and many were found to have both good affinity and satisfactory fastness properties.

"Blocking off," a similar effect to the one observed in dyeing nylon, was noticed on Orlon acrylic fiber. When dyes of various saturation points are mixed together, colors of low saturation point are blocked off by those of high saturation point. Blocking off is either complete when the difference in saturation value is large, or partial when the relative values are closer. Figure 3 shows the effect discussed above. When two colors, both of high saturation point (top left) or both of low saturation point (top right), are mixed, no blocking takes place.

Hudson-Mohawk Section——

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E SZLOSBERG
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a) Orlon 81—all-filament fiber
b) Orlon 41—staple fiber

The dyeing properties of these two types of Orlon acrylic fibers differ to a large extent, and the results obtained on 41-type Orlon could not be reproduced on the 81 type. Comparative dyeings of Orlon 81 and 41 (Figure 1) show that different types of Orlon exhibit different absorption rates relative to acrylonitrile content and degree of orientation. It must be stressed here that the difference in absorption rates between 81- and 41-type Orlon is of a decisive value, and, while the latter produces fast-to-washing shades (including navies, maroons, dark greens and blacks) with reduced copper in the bath, 81-type filament Orlon gives a much reduced exhaustion and wet fastness. At its present stage of development the dyeing of Orlon depends upon adding to the dyebath a reduced copper salt, which is absorbed by the fiber at a rate dependent upon several factors. The rate and the amount of copper absorption will obviously determine the ease or difficulty with which a selected dye or dyes are absorbed.

The rate of copper absorption by Orlon depends mainly on the following factors:

a) pH value of the bath
b) Temperature
c) Time

The pH value of the dyebath is of decisive importance in dyeing Orlon acrylic fiber and thus must be controlled throughout the dyeing operation. There is only a limited range of pH values at which a successful dyeing can be made. When pH value of dyebath is plotted against absorption of copper by Orlon acrylic fiber, it is found that the absorption of copper gradually increases as the pH value rises from 2 to 4, becoming approximately constant at pH 4. When the pH of the bath drops below 2, the amount of copper absorption diminishes considerably, and, in consequence, only surface dyeing takes place. Theoretically, then, the optimum range of pH values is within the limits of 2 to 4. In practice, however, it is found that the reduction of cupric sulfate by hydroxylammonium sulfate proceeds too rapidly if the pH value is allowed to rise above 3, and thus precipitation may occur. The practical methods of keeping pH values within this range will be indicated later.

Figure 2 shows comparative dyeings of Orlon 81 and 41 made at 180°F, 200°F and 212°F. It is evident that the amount of dye absorbed by the fiber (and therefore the amount of copper absorbed) is proportional to the temperature of dyeing. In the case of Orlon 81 the bulk of dyeing takes place at temperatures close to 212°F, and thus actual boiling temperatures are necessary for successful operations.

Time element plays an important part in exhaustion of color. Since the reduction of cupric sulfate to its cuprous form proceeds gradually and the fixation of color on the fiber follows with a time lag, the time required for best exhaustion must necessarily be prolonged.

AFFINITY OF DYES———Among the various classes of dyes that can be applied to Orlon fiber under various conditions of temperature the following are selected:

colors	
basic	chrome
acetate	direct
acid	vat

In this classification the most satisfactory results have been obtained with selected acid colors. In this group there are many dyes with good affinity for Orlon 41, but a limited number producing full shades on Orlon 81. For the latter, at present, only several dyestuffs can be found with satisfactory fastness properties. Among them are the following:

Alizarine Light Blue 2A	Pr 12
Rocceline S	CI 176
Orange Y	CI 151
Chinoline Yellow O Conc	CI 801
Xylene Milling Yellow S	CI 145

* Presented before the Hudson-Mohawk Section in Albany, N Y, on May 2, 1952.

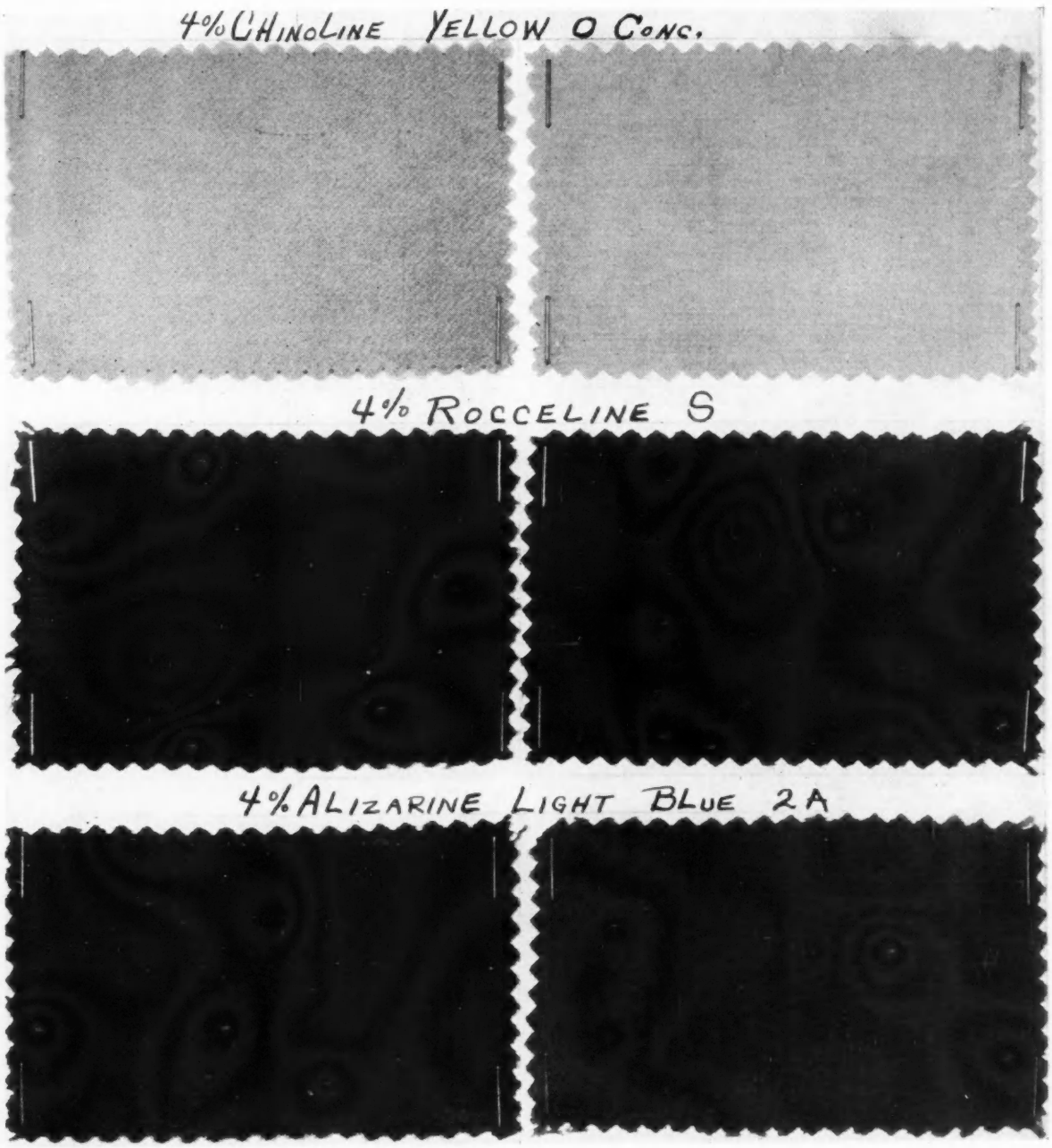

Figure 1
Strength Comparisons
Orlon 41 (left) versus Orlon 81 (right)
Dyed by Drip Method
4% dye
10% cupric sulfate
4% hydroxylammonium

A large number of acid colors were tested on 41-type Orlon acrylic fiber, and many were found to have both good affinity and satisfactory fastness properties.

"Blocking off," a similar effect to the one observed in dyeing nylon, was noticed on Orlon acrylic fiber. When dyes of various saturation points are mixed together, colors of low saturation point are blocked off by those of high saturation point. Blocking off is either complete when the difference in saturation value is large, or partial when the relative values are closer. Figure 3 shows the effect discussed above. When two colors, both of high saturation point (top left) or both of low saturation point (top right), are mixed, no blocking takes place.

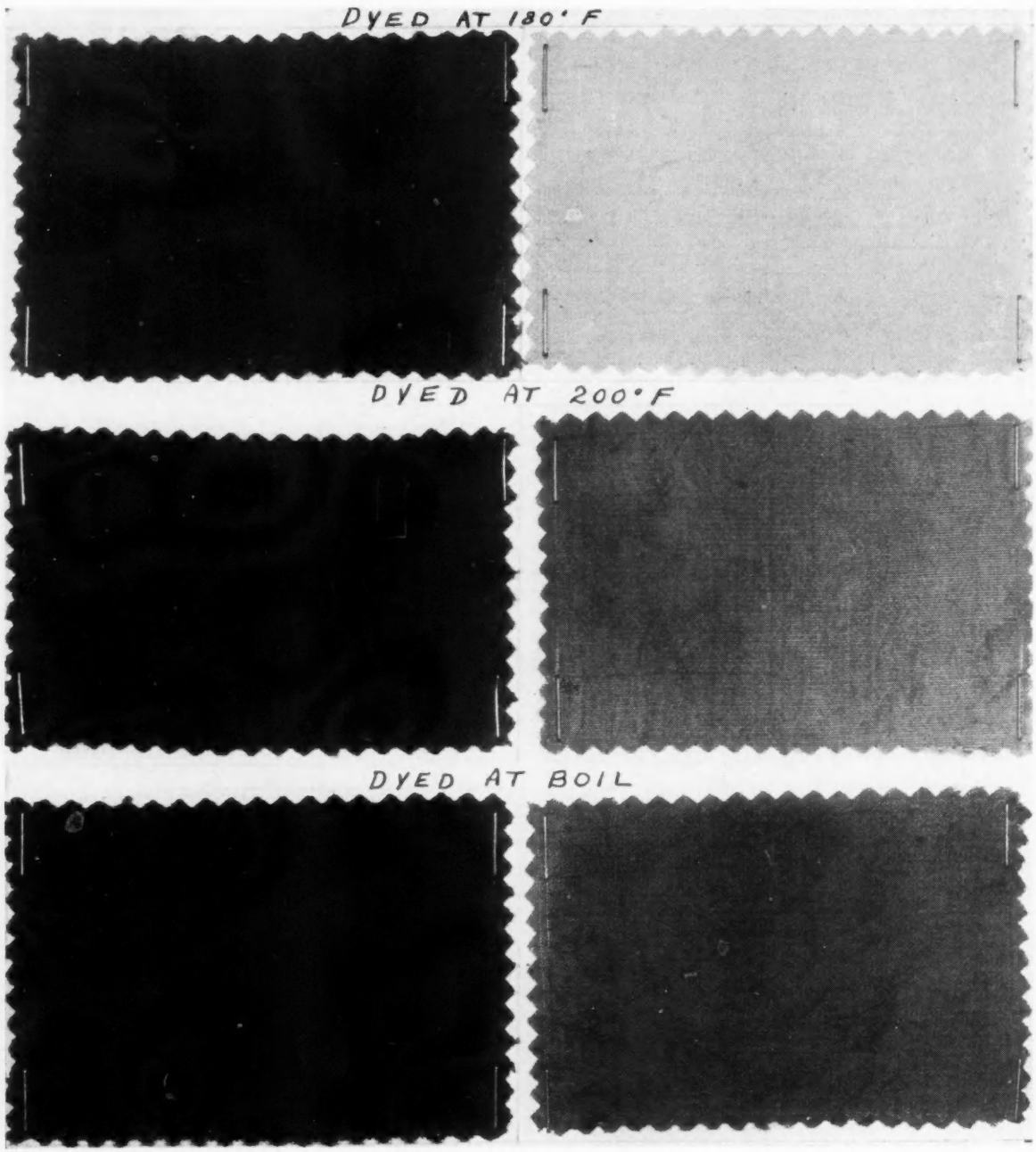

Figure 2
Exhaustion of an Acid Dye at Various Temperatures
On Orlon 41 (left) and Orlon 81 (right)
Dyed by Drip Method at pH 5 (NaOH)
4% Alizarine Light Blue 2A
10% cupric sulfate
4% hydroxylammonium sulfate

When, however, colors of high and low saturation points are mixed together (top middle) the blue element of the mixture (low saturation color) is completely blocked off by yellow (high saturation color). Bottom dyeings show the same effect when mixtures of blue and red are mixed. From these considerations it is evident that the selection of dyes in a mixture must be made according to their exhaustion rates if blocking off is to be avoided. Among the acid colors with the highest affinity for Orlon acrylic fiber are those which have the least number of sulfonic or carboxylic groups in the color molecule. Thus, such colors as Rocceline S or Orange Y and others with one sulfonic group in the molecule exhibit best

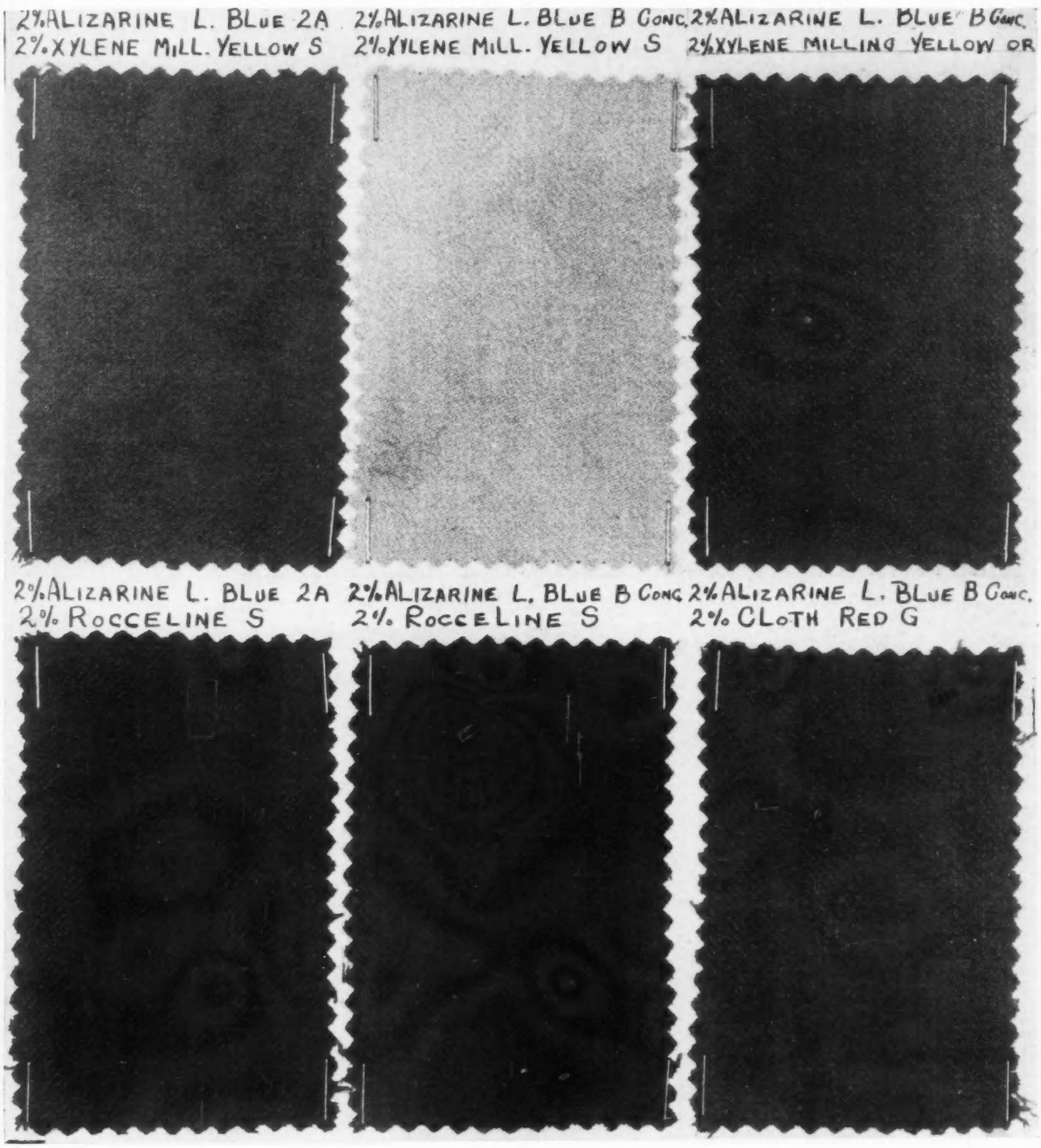
2% ALIZARINE L. BLUE 2A
2% XYLENE MILL. YELLOW S
2% ALIZARINE L. BLUE B CONC.
2% XYLENE MILL. YELLOW S
2% ALIZARINE L. BLUE B CONC.
2% XYLENE MILLING YELLOW OR
2% ALIZARINE L. BLUE 2A
2% ROCCELINE S
2% ALIZARINE L. BLUE B CONC.
2% ROCCELINE S
2% ALIZARINE L. BLUE B CONC.
2% CLOTH RED G

Figure 3
Blocking-Off Effect
Orlon 41 Dyed by Drip Method for 1 Hour at 200°F
dyestuffs
10% cupric sulfate
4% hydroxylammonium sulfate (pH 5, NaOH)

affinity for Orlon acrylic fiber.

REDUCING AGENTS———As stated above, copper is introduced into the bath in its cuprous form. Since cuprous salts are unstabie in aqueous solution, it is necessary to reduce the cupric salt with a suitable reducing agent, which must be powerful enough to reduce cupric salt to its cuprous form. The compound used must be such as not to affect the dyestuffs; the destruction of the latter would of course eliminate all its other advantages. Lastly, reducing agents must possess a rate of reduction comparable with the rate of absorption of copper by the fiber. If rate of absorption is slower than reduction rate, precipitation of metallic

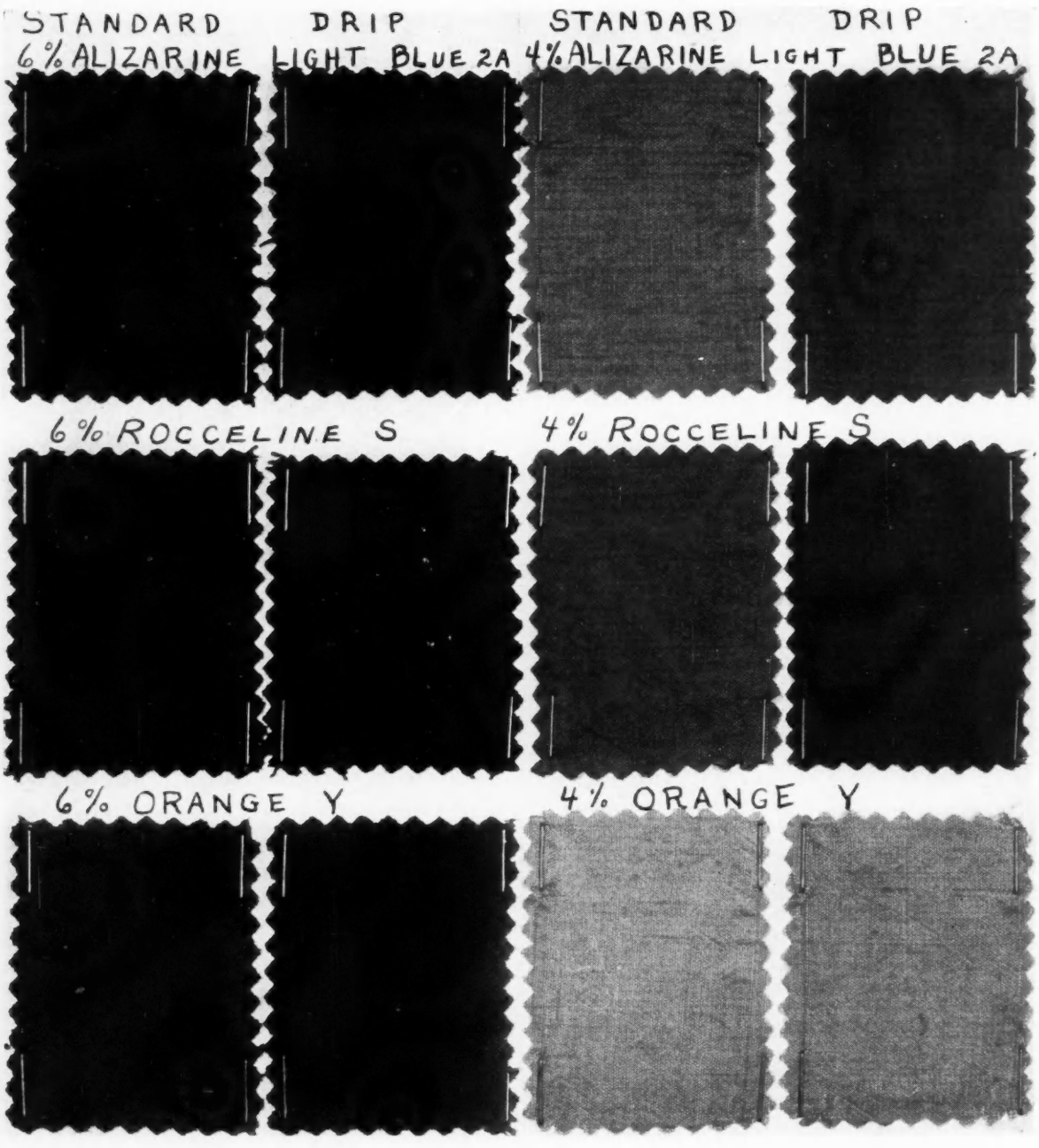

Figure 4
Standard Versus Drip Dyeing Method
Orlon 41 (2 left columns) and Orlon 81 (2 right columns)
Dyed at 200°F and pH 5 (NaOH)
dyestuff
10% cupric sulfate
4% hydroxylammonium sulfate

copper occurs in the dyebath. For these reasons the use of zincformaldehyde-sulfoxylate as a reducing agent was found troublesome and unsatisfactory. Practical demonstrations showed that in many cases its addition to the bath caused precipitation of metallic copper. A more uniform and reliable reducing agent giving better exhaustion values was found in hydroxylammonium sulfate. This compound will effectively reduce cupric salt to its cuprous form at the rate required. There are at present two types of hydroxylammonium salts that can be applied as reducing agents for copper:

1) Hydroxylammonium sulfate $(NH_2OH)_2H_2SO_4$, the aqueous solutions of which give a pH value of approximately

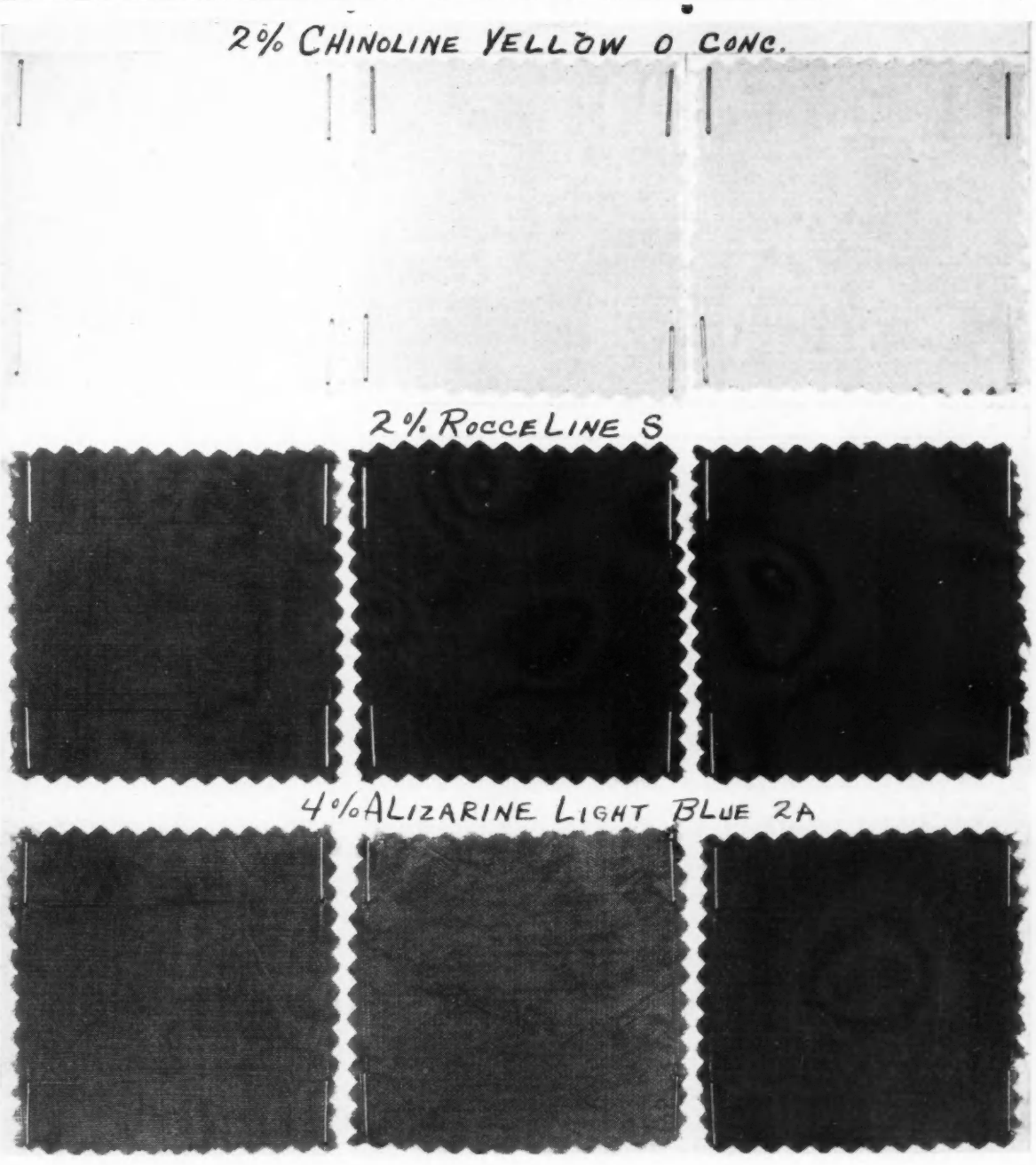

Figure 5
Influence of Swelling Agents on Exhaustion On Orlon by Drip Method
Left column, no swelling agent
Middle column, 0.5% benzoic-acid solution
Right column, 0.5% phenol solution

3.

2) Hydroxylammonium acid sulfate $NH_2OH\ H_2SO_4$, which gives a pH value of approximately 1.5.

As indicated above, it is necessary to control the pH value of the bath and to maintain it within the range of 2 to 3. In order that the pH remains above 2 at all times, the following precautions are necessary:

1) The aqueous solution of hydroxylammonium sulfate should be neutralized to a pH value between 5 and 6 before it is added to the bath.

2) No acid should be added to the dyebath except to correct the condition of water.

3) The use of a buffering compound,

such as monobasic phosphate, is recommended when, for any reason, the pH value tends to drop below 2.

PROCEDURES———A standard cuprous-ion procedure used successfully in the laboratory is as follows:

After being scoured and rinsed, Orlon is treated as follows in cupric sulfate solution at 90°F:

For dark shades, 10% cupric sulfate
For medium shades, 5% cupric sulfate
For light shades, 2% cupric sulfate

At the end of this period hydroxylammonium sulfate previously neutralized to pH 5-6 outside the bath is added, and Orlon is treated for an additional 20 minutes, during which the temperature is raised to 130°F. For one part of cupric sulfate use 0.4 pts of hydroxylammonium sulfate neutralized to pH 5-6 with sodium hydroxide, tetrasodium pyrophosphate, or ammonium hydroxide. Well dissolved dyes are then added, the temperature is raised to the boil in 30 minutes, and boiling is continued for 1 hour. The dyed Orlon is then rinsed, soaped off to remove excess of color, rinsed again and dried. During practical mill runs it has been found that, when the amounts of dyeing assistants were large at the start of the dyeing operation, the reduction of cupric sulfate had a tendency to proceed too rapidly and metallic copper precipitated as the result. This precipitation was originally thought to occur because of faulty neutralization of hydroxylammonium sulfate. It was noted, however, in the course of various demonstrations, that, which ever neutralizing agent was applied (caustic soda, ammonium hydroxide or tetrasodium pyrophosphate), a precipitate was formed and could only be cleared on addition of an excess of sulfuric acid. When, however, sample lots were dyed both on a closed jig and a box, precipitation either did not occur at all or was only slight. It was concluded that, when cuprous-salt concentrations in the dyebath are high, the rate of copper absorption is too low in relation to the rate of reduction. Also, the ratio of one part of cupric sulfate to 0.4 parts of hydroxylammonium sulfate is not maintained when the latter is added to the dyebath, since the contact between the two assistants takes place not over the whole of the liquid but in the limited area of addition. The latter remark applies mainly to box dyeing.

DRIP METHOD———To overcome these problems we now recommend a modified copper ion method, hence referred to as the "Drip Method." 10% of cupric sulfate and 4% hydroxylammonium sulfate (pH 5-6) are reacted cold outside the bath and the resultant solution diluted to a suitable volume. 1/10 to 1/5 of this solution is added to the dyebath cold, the temperature is raised to 130°F in 10 to 15 minutes, well dissolved dyes are added at this point and the temperature is slowly raised to the boil and boiling is continued for 30 minutes. At the end of this period the remainder of the cuprous salt is added or *dripped* into the bath over 30 minutes, while boiling is continued. When the balance of cuprous salt has been added, the dyebath is allowed to boil an additional 30 minutes. It is then slowly cooled, the Orlon rinsed, soaped off, rinsed again and dried. This modified procedure gives better exhaustion, more uniform dyeings and is particularly valuable when dyeing large quantities of Orlon. Figure 4 shows the comparative strength of three colors dyed by standard and drip methods on Orlon 81 and 41.

The procedure, as described above, will produce fast-to-washing shades on 41-type Orlon. It can also be applied to all-filament 81-type Orlon. In the latter case, however, the exhaustion of color is considerably reduced, and the dyer may find it difficult to obtain deep shades with satisfactory wet fastness. In such cases it is advisable to introduce into the bath a chemical swelling agent along with cuprous salt in order to promote penetration.

The compounds found to give required results are as follows:

phenol (carbolic acid)..... 0.5% solution
benzoic acid ..... 0.5% solution

With the exception of phenol, swelling agents should be added to the dyebath at the boil inasmuch as their solubility in cold water is limited. Since phenol dissolves readily in water (in concentrations required for optimum processing) it can be added to the dyebath before the cupric salt. Figure 5 shows the effect of swelling agents on exhaustion of acid colors on Orlon 81.

PRECAUTIONS———Finally, a word about precautions to be observed in dyeing Orlon acrylic fiber.

1) The cuprous-ion method may prove detrimental to stainless-steel equipment of type 304. Type 316 is not affected.

2) When dyed in the presence of phenol, Orlon should be well scoured with soap or synthetic detergent at the completion of the dyeing operation. Contact of phenol with the skin should be avoided.

3) Hydroxylammonium sulfate should be neutralized with constant stirring.

4) Some types of water, particularly those softened by the Zeolite process, contain enough alkalinity to cause partial precipitation of cupric salt. In order to counteract this, it is advisable to acidify such water with sulfuric acid until a clear solution of cupric sulfate results.

It must be stressed that the results contained in this paper should be considered only as a report of the progress of our findings as to current problems in dyeing Orlon acrylic fibers. It is therefore quite possible that our statements might lose their validity in the light of future developments.

SUMMARY

Orlon 41 and 81 acrylic fiber are dyed upon the introduction of cuprous salt into the bath.

The success of dyeing depends upon the pH value of the bath, the temperature of dyeing, the time of dyeing, and the selection of reducing agent and dyes.

Controlled addition of cuprous salt (drip method) improves exhaustion and uniformity, particularly when large lots are dyed.

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2) T A Feild, Jr and G H Fremon, "The Copper Technique for Dyeing Acrylonitrile Fibers," *Textile Research J* 21, No 8, August, 1951.

National Officers to be Guests at Next Philadelphia Section Meeting

THE next meeting of the Philadelphia Section wil be held at the Penn-Sheraton Hotel, Philadelphia, Pa, on September 12th, in conjunction with the regular September meetings of the Council and General Research Committee. National officers and committee members have been invited to be guests of the Section.

Arthur W Etchells, Hellwig Dyeing Corporation, will be host and chairman for the evening session, which will be preceded by dinner at 7 pm. C Norris Rabold (The Erwin Mills, Inc), AATCC president, will discuss in detail the aims, objects and activities of the Association in "The President's Message". Other speakers will include Leonard S Little, Chairman of the General Research Committee, and Dr H W Stiegler, Director of Research. Plans also call for a showing of a film on tests methods by the J C Penney Company.

1952 OUTING PHOTOGRAPHS WESTERN NEW ENGLAND SECTION

WALLINGFORD COUNTRY CLUB WALLINGFORD, CONN.

WNE Chairman Rudolph C Geering (left) and Joseph F Melody, Outing Chairman

Photos by A Henschel and A H Rant

Rhode Island Section ——

STATIC ELECTRICITY IN TEXTILES*
A Stepchild of Modern Research

SIDNEY M EDELSTEIN
Technical Director
Dexter Chemical Corporation

S M Edelstein

The author again effectively employs the question-and-answer method to present his views with respect to the use of a type of textile chemical specialty, namely, antistatic agents. He defines and explains static electricity and points out the difficulties it causes in both the manufacture and use of textiles. The new, wholly synthetic hydrophobic fibers aggravate the problem.

In the manufacture of textiles static has been controlled by humidification of mills, by dampening of fibers, by application of hygroscopic substances, by application of electrolytes, such as salts and sulfonated products, by grounding of textiles and machines, and by ionization of air at trouble spots either with radio-active substance or by electrical discharge. The author emphasizes the advantages arising from the application of specially compounded chemical antistatic agents to textiles for the control of static. These not only may continue their effect on the textile until it reaches the consumer and contribute to finish, but they can be so designed as to possess some fastness.

INTRODUCTION

EXACTLY two hundred years ago this very month, the scientific world was agog over the latest investigation into the fundamentals of static electricity proposed by Benjamin Franklin———the drawing down of electricity from the clouds.

Today our textile world is agog over the problem of static electricity in textiles, and yet our fundamental knowledge of static electricity is not much greater than it was during Franklin's time. The recent introduction of many new synthetic fibers has made the static problem in textiles more acute. We have been forced to begin at least an attack on the problem from several directions. While some progress has been made toward a better understanding of the problem, and while we already have some practical means for controlling static electricity to some extent, much remains to be done. Tonight we propose to answer a few of the many questions posed by the problem of static electricity.

I. *What is static electricity?*

It is surprising how difficult it often is to define a term which we use constantly and glibly. "Static electricity" is no exception. I believe most of us have confused the effects of static electricity with the actual thing itself.

The word "static" is very definite, and the definition given to us in the dictionary is quite satisfactory. The dictionary defines the word "static" as pertaining to bodies or forces at rest. Therefore, static electricity is electricity at rest. But what of the word "electricity." Our dictionary doesn't give a satisfactory answer and neither do our textbooks. Suppose we move back into the past in order to find out where the word "electricity" came from.

As long ago as 600 BC Thales, a Greek, knew that amber when rubbed would attract small pieces of paper and other objects. The Greek name for amber was "elektron" and the Latin "electrum." When Sir William Gilbert about the year 1600 found that many other substances beside amber have the property of attracting bits of paper when rubbed, he gave the Latin name of "electrica," meaning amber-like, to such materials. Now, the English name corresponding to the Latin "electrica" becomes "electrics." The word "electricity," however, was first used by Sir Thomas Brown in 1646. He used this term to mean the active principle, or force, which appeared in electrics after they were rubbed, and which force causes attraction for bits of paper.

Today, of course, we have various theories which explain this force on the basis of atomic and molecular configuration, but these are only theories. We still have no better definition for "static electricity" than that which Brown put forward 300 years ago: namely, "static electricity" is that force which causes the attraction of small bits of paper to an object after the object has been rubbed. We must remember that this force may be generated by many other methods and may be evidenced by other phenomena, but it is still the same force.

II. *How does static electricity develop in textile materials?*

Although the fundamental explanation as to how static electricity can be generated in nonconductors is quite complex, in textiles there are only three mechanisms of importance. The first important mechanism is *double-layer separation.* When any two neutral dissimilar substances are brought into contact, electric charges occur in pairs of positive and negative kinds. Negative charges, that is to say, electrons, migrate to one substance making that substance negatively charged; whereas the other substance, having lost electrons, becomes positively charged. Now, the positive charges on the one substance are exactly equal and balance the negative charges on the other substance. The number of charges developed in the substances depend on the chemical nature of the substances, the extent of area in actual contact, and the pressure between the two substances. We only become aware of the presence of these charges when the substances are separated. For then, if one substance is a nonconductor, such as a textile fiber, the charges which it held become entrapped, and the fiber becomes negatively or positively charged. If the other substance is a conductor, then its charges are almost immediately dissipated, and this substance becomes neutral. If the second substance, however, is also a nonconductor, it then shows a charge on separation from the textile fiber, which is equal to but opposite in sign to that on the fiber.

The second important mechanism for the generation of static electricity is friction.

* Presented in Providence, R I, before the Rhode Island Section on May 23, 1952.

If two neutral dissimilar substances are rubbed together, negative charges are rubbed off one substance and are taken up by the other. Upon separation of the two substances after rubbing, the nonconductor, or textile fiber, entraps its charges and becomes either positively or negatively charged. If the other material is also a nonconductor, it will have an equal but opposite charge.

There is a question among scientists as to whether the mechanism for forming static electricity by friction is really different from the double-layer mechanism. Some think that friction simply promotes closer contact between the two surfaces. Others believe that there is some fundamental effect caused by friction which is in addition to the effect caused by simple contact of the surfaces.

The third mechanism is important only in special cases. In a flame, lighter or more diffusible particles of carbon and other substances have a charge opposite to that of the heavier particles. When a nonconductor, such as a textile, is held close to a flame, the heavy charged particles tend to attach themselves to the textile, and the textile becomes charged, or develops static electricity.

With an understanding of the mechanisms whereby static electricity may be generated in a nonconductor, such as a textile fiber, it becomes easy to visualize how such generation may take place in blending, spinning, weaving, and drying textiles, and also in the use and wear of fabrics. In other words, at every point where a textile touches another substance, static electricity may ge generated. The static may not always be great enough to make itself known, but it's always there, a potential source of trouble.

III. *What troubles are caused by static electricity in the manufacture of textiles?*

By this we mean, "what troubles could static electricity cause if it were uncontrolled?" Of course, for many years we have been controlling static in some textile processes without realizing it. The use of oils on cotton and wool in carding and spinning has not been for lubrication so much as for static control. The need for high-humidity conditions in cotton weaving has not been required for conditioning the cotton so much as for controlling the static problem.

The effects of static electricity in the processing and manufacture of textiles are mostly undesirable effects. How undesirable these effects are will depend on many factors, one of the most important of which is the chemical nature of the fiber. As our fibers become more nonconducting, and as they become less moisture-absorbing, each potential source of trouble due to static becomes greater. That is why when we deal with the new synthetic fibers, every potential source of trouble from static becomes an actual source of trouble unless we take special steps to control the static. In the case of natural fibers, such as cotton and wool, many of the potential sources of trouble never actually break out into real trouble, but when conditions are right, trouble will be there.

Recently a writer (*1*) has pointed out that static electricity could cause four major problems in textile manufacturing:

1) Reduction of output.
2) Lowering of quality.
3) Increase in waste.
4) Hazards to personnel.

Each of these major problems is potentially present in most textile operations. They may exist in blending and carding down through drawing, sizing, knitting or weaving, and even in the final operations of inspection and packing.

In the carding of woolen, cotton and synthetic fibers, static charges can cause the fibers to load on the cylinders, and may even prevent the fibers from forming a web.

During spinning, static electricity may cause the fibers to repel each other, thus producing uneven yarns. The yarn may be attracted to various parts of the machinery and thus broken ends may result, or it may be repelled from the guides, causing loops and breaks.

During sizing, the yarns may repel each other to give beams with the yarns badly aligned. During weaving, static electricity may cause lint and dirt to be attracted to the yarn, and this may accumulate on the heddles and drop down in one mass to be woven into the goods, thus causing seconds. Also, warp yarns may repel each other to give an uneven weave, and static electricity may even prevent filling yarns from coming off free from the shuttle, causing breaks and loom stoppage.

Finally, at the end of wet finishing, the fabric may emerge from the dryer with enough static electricity to resist folding, and electrified enough to attract lint and dirt from the air or floor.

The potential troubles due to static in textile manufacturing are almost endless in number. As we increase our operating speeds, and particularly as we use more and more of the new synthetic fibers, these potential troubles will become real ones.

IV. *How does static electricity in textiles affect the consumer?*

The effects of static electricity for the consumer run the whole gamut from the commonplace to the unusual. Perhaps one of the most unusual cases was that recently reported by one of the large department stores, wherein they cheerfully refunded the money to a lady who had purchased a blanket made of one of the new acrylic fibers. She was fond of listening to the radio in bed, snuggled up warm within the blanket. Everytime she moved, static electricity was generated within the blanket and spoiled her radio reception, at least, so she said. Unquestionably, the public has really become conscious of the static problem in the newer fibers.

Another static story is that reported by a men's wear store of a customer who had bought a suit specially made of one of the new synthetics for summer wear, when conditions for static generation are at their poorest. This particular customer had chosen to wear the suit, however, on a cool, dry Fall day. The store reported that the gentleman came running into the store shouting, "Get it off; get it off." The suit was clinging and twisting around him like some evil spirit. Of course, all that would have been needed to complete the picture would have been a discharge of St Elmo's fire from the man's head, and this would have given emphasis to a report that a man from another planet had landed from a flying saucer.

Now, most of the consumer problems due to static are much less exciting, and not too spectacular, but they are very important, nevertheless. Some have been with us for a long time, such as the shock we often get from a carpet on a cool dry day. But, the static problem from the consumer viewpoint has only become acute with the widespread use of wholly synthetic fibers, such as nylon, and more recently with the employment of Vinyon polyvinyl fiber, Dacron polyester fiber, Orlon polyacrylic fiber, Dynel, and other fibers based on synthetic plastic materials.

These problems fall into certain distinct groups, which are common to all the fibers. First, there is the effect of static electricity in causing clothing to cling to the body under certain conditions, or to be repelled from the body under others. The important draping qualities and appearance of the fabric, or garment, are often completely ruined by the effect of static. The uncomfortable feeling to the wearer of the garment is obvious.

Unfortunately, a textile charged with static electricity has the power of attracting uncharged particles. Therefore, the static problem in the new synthetics has added the problem of dirt attraction. This is not only a problem for the store in displaying an article made of one of the new synthetics, but it is certainly an unpleasent condition for the consumer who buys a suit made of a new synthetic fiber because of its stain-resisting properties only to find that his suit attracts all the black soot and lint in the neighborhood.

Several of the synthetic plastics and

fibers have been used to make so-called allergy-free pillow cases and garments because of their clean, lint-free nature. But, if these articles under certain conditions become easily charged with static electricity, the attraction of almost invisible particles of dirt and spores and bacteria completely defeat the original purpose behind their use.

Finally, there is the special case where fabrics of certain of the new synthetics are made into garments for use in refinery and chemical plants. Their high resistance to chemical attack has offered a wonderful new protection to the worker. Their use, however, has also offered a potential explosion hazard because of their ability to build up static charges to the point where explosions of solvents may take place.

Now, all of these problems offer a real challenge to each of us. For the new synthetics have so many advantages that their use will increase in spite of static.

TABLE I

POSITIVE END

glass
human hair
nylon yarn
nylon polymer
wool
silk
viscose rayon
cotton
paper
ramie
steel
hard rubber
acetate rayon
synthetic rubber
Orlon acrylic fiber
Saran
polythene

NEGATIVE END

V. *What are the conditions for preventing static buildup in textiles?*

There are two obvious answers to this question and these are as follows: either neutralize the charge as soon as the charge is formed, or conduct away the charge as soon as it is formed.

Since the matter of conducting charges away will be dealt with in our next question, we shall only discuss now methods for neutralizing the charges as soon as these charges are formed.

Many investigators have studied the charges developed when common solid substances, including various fibers, are rubbed together. They have arranged these substances together into so-called electrostatic series. These series are made up so that when any substance is rubbed with a substance lower in the series, the one lower in the series will develop a negative charge and the one higher will develop a positive charge. These electrostatic series are in fairly good agreement among investigators. A typical electrostatic series, recently published, lists the items as in Table I.

As an example of how this series works, suppose we choose nylon. From this series, if nylon comes in contact with glass, it will develop a negative charge, but if it comes into contact with rubber, steel or any of the other substances below it in the series, it will have a positive charge.

Therefore, the one obvious way for neutralizing the charges built up in nylon would be to impregnate the nylon with some material that is very much lower in the series, and which, when in contact with rubber, steel or other materials, would develop a negative charge. In this way, suppose the treated nylon yarn or fabric were passed over rubber or steel rolls, or over both. As a positive charge developed in the nylon, a negative charge would develop in the impregnating material, and theoretically these would neutralize each other. Now, theoretically such a mechanism is sound. Practically, the actual use of this mechanism is only in its infancy, but it will become more important pending our increase in knowledge of the subject.

The other mechanisms for preventing build-up of charge involves bombarding the textiles with charges opposite to that of the charges being built up in the textile. This mechanism is theoretically simple but difficult to control.

First, radioactive materials can be used as a source of either positive or negative charges. Since certain radioactive materials cause ionization of the air into positive and negative particles, it therefore only becomes necessary to have a radioactive substance close enough to the textile, and sufficient strength to furnish enough ions. Whether the fabric tends to develop positive or negative static electricity is unimportant for the charges developed in the fabric will draw to itself the oppositely charged ions in the air and thus become neutralized. While, theoretically, radioactive material could be incorporated in the fiber or in the fiber finish, it is still a moot question as to whether the presence of enough radio-active material in a textile, from the static-prevention viewpoint, would be strong enough to cause harmful effects to the users of the textile.

Finally, it is theoretically possible to blast the textile fibers with ions from high-voltage discharge sources, so that these ions will have a charge opposite to that developed in the textile, and thus the charges in the textile will be neutralized and no charges can be built up. Obviously, these theoretical mechanisms, while interesting, can only be brought into use under certain very special practical conditions.

VI. *Under what conditions can static electricity leave a textile?*

If we cannt prevent the build-up of a charge by any of the means mentioned above, there are mechanisms for the removal of this static electricity from the fabric almost as soon as the charge is built up, or at least fast enough so that no harmful effects are noticed.

There are obviously only two paths whereby static electricity can be conducted off of the textile. First, let us consider grounding. The ground or earth may be considered to be an infinite reservoir of positive and negative charges. If a charged fabric is grounded, it must sooner or later lose its charges. Unfortunately, however, textiles are inherently poor conductors, and those that develop static electricity to the greatest extent are the most nonconducting. Obviously, while grounding a textile is a theoretical technique for removing charges, it can mean nothing unless the conductivity of the textile is so changed by treatment so that it can quickly conduct a charge to the ground.

Secondly, we have the mechanism of discharge of static electricity to the atmosphere. This is related to the humidity of the atmosphere, the presence of gases and other substances in the air, and, most important, to the surface conductivity of the textile. This latter property will be discussed later when we discuss chemical antistatic agents.

Suffice to say, paths for removing charges from textiles are available. Our problem is to make use of these paths.

VII. *Can the static problem be controlled by humidity regulation?*

Our first answer to this is, "Yes," for obviously cotton has been spun and woven under humidity conditions high enough to control the static problem for a very long time. If our question asks, "Is humidity regulation the best method for static control?", and particularly if it asks, "Is humidity regulation satisfactory for the control of static with the new synthetics", the answer is "No".

First, we must examine the theoretical part that humidity or moisture in the air can play in its effect on static electricity. To examine this problem more clearly we must separate our textile fibers into those that are hygroscopic and those that are not.

In the case of the fibers that are hygroscopic, the amount of moisture in the air will govern the amount of moisture in

and on the fibers. This moisture itself, in contact with fibers, has a definite amount of conductance, which assists in the removal of electric charges to ground and to the atmosphere. There is an additional effect with higher humidities, for a high humidity actually improves the ability of the atmosphere to take up electric charges from the textile.

Now, in the case of nonhygroscopic fibers we have only the effect of high humidity in improving the power of the atmosphere for conducting away charges. There is a little improvement in surface conductivity of these fibers unless the humidity is so high that the point is reached where there is actual condensation of moisture on the fibers. Under such conditions the presence of foreign matter on the surface of the fiber in contact with the water may actually add some conducting power to this surface-condensed water so that there is some surface conduction. This conductance, however, is very small compared with the surface conductivity changes encountered with the hygroscopic fibers.

Finally, there is a certain range of humidity where a reduction below this range actually reduces the amount of static electricity found, instead of increasing it as normally expected. This unusual condition is encountered under very low humidity ranges, which are not often found in practice.

The disadvantages of high humidity as a means of static control are obvious. In the case of the new nonconductive fibers, high humidity is not very effective, because these fibers also have low moisture regain. In the case of the natural fibers, however, which have a higher moisture regain, high humidity conditions are capable of controlling the static problem, but these high-humidity conditions are often unpleasant for the working personnel and make ideal conditions for the corrosion of expensive machinery and parts. Finally, humidity conditions must generally be kept within a narrow range even for the hygroscopic fibers in order to obtain satisfactory results. As we shall see later, there are other means which are more satisfactory for the control of static.

VIII. *What is the place of electrical devices in the control of the static problem in textiles?*

As we have already pointed out, static electricity is a problem both in the manufacture of textiles and in their use. Electrical devices must, because of their very nature, be considered only in connection with textile manufacture. There are only three basic electrical devices which, up to the present, have been employed in an attempt to control the static problem in textile manufacture. Each of these basic types may take many different commercial forms.

Let us first look at grounding devices. It is possible to ground each and every machine that comes in contact with a textile more or less effectively. This will prevent a build-up of high charges in the machine or in the part touching the textile, but the charge built up on the textile is essentially unaffected. The grounding of machinery may be of course a useful technique in protecting personnel.

Grounding devices may actually be made to contact a moving textile in an attempt to carry off the charge built up on the material. Metal brushes or metal foil connected to ground may contact the moving textile at key points with the thought that the charge on the fibers will be drained off. Unfortunately, however, the amount of charge that may be drawn off by such devices is not large enough usually to discharge completely the static electricity entrapped within the fibers. Such devices, however, are often useful to prevent extremely high voltage build-up at points where explosion of vapors could take place. A very definite disadvantage in such grounding devices is their short life if the device in contact with the fabric is very light. If we use a heavy brush or a contact with a longer life, we may cause damage to the textile.

The use of radio-active substances has been discussed already in theory. Practically, such substances have been employed to eliminate or control the static built up on moving textiles at key points in their manufacture. These devices usually consist of bars containing the radio-active substances, which are suspended above or below the moving textiles. The outstanding disadvantage of this type of device is that when static conditions are very severe or when the textile moves at high speeds, the textile cannot be bombarded with enough ions to discharge the static completely unless prohibitive concentrations of radioactive materials are employed.

Acting in the same manner as the radioactive static control device, that is to say, by ionizing the air and bombarding the textile with ions, is the high-frequency or high-voltage discharge bar (*1*). This device can be placed at key spots where static elimination is most desirable. The difficulty with such devices is that, without careful control and adjustment, the moving textile may even be recharged where intense conditions are required. Also, it has been reported that maintenance costs for such devices are often excessive.

A recent modification of the high-voltage discharge device has been the so-called controlled diffuse discharge unit, which is also based on the principle of bombarding the textile with ions that have been produced in the air by high voltage. In this device it is said that the moving textile is bombarded with ions having a charge opposite to that on the textile, and that, as soon as the charge on the textile has been neutralized, the discharge from the bar is instantly diverted to collecting targets built into the static control bars. In this way there is no possibility of recharging the textile.

The obvious disadvantages of all of these devices is that they can only be used at key spots. As soon as the textile has passed the spot and has contacted a new surface, static electricity is again generated with all its attendant troubles.

IX. *What is the place of chemical antistatic agents?*

In theory the use of a chemical antistatic agent on a textile is an ideal method for controlling static electricity. As we have already seen, electrical devices can only be used at certain points past which the textile travels, and we have seen that humidity can only be used to control static under certain conditions, and satisfactorily only with the older fibers. None of these devices or methods previously discussed can be of much use in controlling the static problem for the consumer. A chemical antistatic agent, however, can be put on the fiber, and it will travel along with the textile to do its work at every point. Also, from the consumer standpoint the use of chemical antistatic agents is the only possible answer to static control in textiles.

This does not mean that there are no problems in connection with chemical antistatic agents. There is the problem of where to apply the antistatic agent. Should it be easily washed off, or should it be fast. What about the effect on the hand, on strength and on the appearance of the fibers. We often must take into consideration its compatibilities with many materials. We must think about whether it will affect the light-fastness of the colors, or whether it will develop an odor. In fact, use of chemical antistatic agents poses many problems. But, only by the use of chemical antistatic agents can we look for a real solution to the problem of static control in textiles.

X. *How does a chemical antistatic agent work?*

All of the mechanisms whereby a charge can be neutralized before it builds up and the ways whereby charges can leave fibers may be taken advantage of in the use of chemical antistatic agents. This does not mean that all chemical antistatic agents make use of all of these means, but, theoretically, they could.

The mechanism whereby most chemical antistatic agents do their work is in the field of conductance. Now, even if an antistatic agent also makes use of other processes, it always has some effect in improving the surface conductivity of the fiber and thereby in helping charges to go more readily to ground or to the atmosphere. Any surface-active agent that forms ions in solution when placed on the surface of a fiber, even in minute amounts, must improve the surface conductivity of the fiber. On this basis, a film of soap, sulfonated oil or synthetic detergent will improve the surface conductance of fibers and will theoretically assist in the removal of static. It must be borne in mind, however, that, while most chemical substances applied to a fiber increase the surface conductivity of the fiber, some chemicals are many times more effective than others in this respect. This is due to some fundamental relationship not understood and is only partially related to the conductivity of the chemicals themselves in solution. There are indications, however, that the improvement in surface conductivity caused by chemical antistatic agents is related to the type of film the agent forms on the fiber, and to the way in which the molecules are oriented on the fiber.

Another way whereby a chemical antistatic agent can do its work is in the matter of hygroscopicity. This is particularly important with the new low-regain synthetic fibers. We have seen that the presence of moisture on the surface of a fiber assists in the lowering of surface resistance and hence improves the rate of charge removal. Hygroscopic antistatic agents thus can do part of their work by attracting molecules of water to the surface of the fiber. This water also serves to activate further the chemical antistatic agent by partial solution of the active groups of the agent in the water.

Thirdly, a chemical antistatic agent may do its work by developing a charge opposite to that formed within the fiber itself when the treated textile is rubbed or comes in contact with another substance. This mechanism seems to be related not only to the chemical nature of the antistatic agent but also to the orientation of the molecules of the agent in the film and to the nature of the film.

A chemical antistatic agent can thus do its work by any one or all three of the above-mentioned ways. It appears, however, that in order to be really effective, an antistatic agent should best act to some extent in all three ways.

In passing we must not neglect to mention the possibility of the incorporation of radio-active elements within the molecule of the antistatic agent itself, but this is really something for the future.

XI. *What is the difference between a finishing agent with antistatic properties and a straight antistatic agent?*

This is like asking what is the difference between the general practitioner and the specialist in the field of medicine. As we may well expect, many finishing agents of themselves have some antistatic properties. Many finishing agents contain or consist of sulfonated oils or fats or soaps. Many contain salts. All of these ion-forming compounds must increase the surface conductivity of fibers to some extent. If the finishing agent is hygroscopic, it also must have some antistatic properties. Finally, if by any chance the finishing agent doesn't have any ion-forming or any hygroscopic agents, a hygroscopic agent can be added or a sulfonated oil or similar ion-forming material can be added, and we have a finishing agent with antistatic properties.

The true antistatic agent is designed for one special job, that is, its antistatic properties are developed to the fullest. While antistatic agents may have softening properties or some other properties that are desirable, their antistatic properties are the main thing. In general, a true antistatic agent will have several times the antistatic powers of an equal weight of a finishing agent with antistatic properties.

For example, in order to obtain the required amount of static protection with a well-advertised and so-called antistatic winding oil, we may have to use 8% on the weight of the fiber, whereas ½% on the weight of the fiber of one of several good true antistatic agents will do the job. Of course, the reverse conditions could be true if lubrication were the main problem and static control only of minor importance. We might need only ½% of the lubricating oil and 8% of the antistatic agent in order to get proper lubrication results.

Unfortunately, because of a lack of a true understanding of the static control problem we find people offering ordinary finishing agents with some antistatic properties as straight antistatic agents. Also, we have found people confusing softening or lubricating problems with static problems and vice versa. An understanding of the difference between the two agents, however, should prevent such confusion. If our main problem is static control, then we need a straight antistatic agent. If the main problem is lubrication or finish and static control is of minor importance, then a finish or lubricant with antistatic properties is recommended.

XII. *Is there one best antistatic agent?*

If this question asks if there is one best antistatic agent on the market today, the answer is "No." If the question asks if we can set up the requirements for an ideal antistatic agent, then the answer is "Yes." If the question asks, however, if we are likely to have an ideal anti-antistatic agent in the foreseeable future, the answer is "Maybe." Let us evaluate each of these three answers.

First, let us look at the antistatic agents available today. An apt quotation is, "Many are called but few are chosen." Actually, nearly every manufacturer of chemicals for the textile industry is offering at least one antistatic agent. Many offer several. Although nearly all finishes have some antistatic properties, as already explained, only a handfull of the agents offered today are true antistatic agents with antistatic properties developed to a high degree.

Among this handful of true antistatic agents none are truly permanent on all fibers, and hence miss the ideal in this respect. Some, however, are more permanent than others. Among this handful of true anti-antistatic agents, I know of only one substantive to Dacron polyester fiber. Several of the antistatic agents among this handful affect the lightfastness of colors and develop yellowing on storage of the goods. Among this small group, some work well on nylon but are ineffective on some of the other fibers. Some of these true antistatic agents have excellent properties in themselves but are not compatible with other finishing agents.

To quality our first answer to the question, we must say that there is no one best product available today, but there are a few true antistatic agents that are very good in many respects and can be used to alleviate many of the static problems in textiles.

To set the requirements for an ideal antistatic agent is not too difficult. First, and foremost, the ideal antistatic agent must have an enormous amount of antistatic properties bound up in each molecule. Therefore, it would have to make use of all of the various mechanisms we have discussed for preventing statis build-up as well as for conducting the static away. This product would have to work and be effective in small amounts on all fibers. It should be permanent to washing and dry cleaning. It should not have any color nor have any effect on the color or the lightfastness of dyestuffs. It should not change the hand or feel of the fibers. In other words, except for its antistatic properties it should act like the little man that wasn't there.

The 'maybe' I have given to the third answer above is simply to point out our goal. Perhaps the ideal will never be reached, but it shall be approached. The concept of a true permanent antistatic agent is not beyond the realm of reason. Increased antistatic properties are certainly conceivable as we learn more about the mechanism of generation and of removal of static from textiles and the relationship of these to chemical structure. Today we have some good antistatic agents. Tomorrow we shall have better ones.

XIII. *Can the efficiency of antistatic agents on textiles be determined by laboratory instruments presently available?*

This question is hard to answer without hedging. There are many instruments being used which do give some information about some of the antistatic properties of a textile. They don't tell the whole story, and in most cases they don't even tell an honest story as far as the practical performance of a treated textile is concerned.

There are two basic types of measuring instruments in use today for determining the effect of antistatic agents on textiles. One basic type measures the charge generated by friction on the treated textile, and the other basic type measures the conductivity or charge leak-off rate of the textile. In many cases the machines are devised to make both kinds of measurements, and in some cases the measurement made is a combination of the two.

The types of instruments in use today are similar in principle and differ mainly in their mechanical design and in the method of charge measurement. Most of these machines operate under controlled conditions of humidity and temperature. All of these machines attempt to cause frictional contact between the test sample and another substance, which is usually hard rubber, wool, felt, glass, porcelain or some other substance. The sample may be pressed under fixed or variable pressure against a moving wheel, drum or disk of hard rubber or other material, or, in certain cases, the sample may be mounted and revolved against the frictional element. The charge built up is measured by an electrometer, a vacuum-tube voltmeter, an electrostatic voltmeter or a generating voltmeter.

There are great differences between the artificial conditions set up in such a machine for generating static and the conditions under which static is developed naturally in practice. First, let us consider the frictional element itself. In many laboratory machines this element is generally only one substance, hard rubber, but at best, may include one or two other frictional elements. In the manufacture of textiles, however, fibers may contact such surfaces as wood, steel, hard rubber, stainless steel, copper, plastics and a host of other substances. In the movement of textile material through a plant, first one substance is contacted and then another, and usually each substance is only contacted with the textile for a very short time. We have already learned that each substance has its own position in the electrostatic series and therefore may generate a positive charge or negative charge with the textile depending on its position in this table. How could the charges built up by contacting the sample over and over with one or at best two substances in the test machine be related to the charges built up by momentary contacts with the myriad of substances that a textile encounters in a plant? These same discrepancies must exist in relating the test results of such machines to the static effects obtained when a woman puts on a pair of nylon stockings or when a man wears a Dacron sweater.

The relation of the laboratory value of electrical conductivity of a sample treated with antistatic agents to practical results is also fraught with danger. In the first place, most conductivity instruments measure the time it takes for a certain high charge, which is usually the charge built up by the frictional machine, to drop to a half value. But we know that the charge under such conditions is not related to the amount of charge which would be present in a fabric under practical conditions. The rate of transference at one voltage is not necessarily related to the rate of transference or conductance at some other voltage.

While several other sources of error in present laboratory instruments could be discussed, such a discussion would be too lengthy for us here tonight. Suffice to say that in the opinion of our technical staff, and in the opinion of several experts who have investigated the subject, there is no instrument described in the literature, or of which we have knowledge, which can accurately or even reasonably foretell the actual performance properties of a treated textile under plant or consumer conditions from the viewpoint of static development.

XIV. *How do we recognize our textile problem as a static problem?*

The majority of cases where static is the cause of trouble are generally easy to recognize. Sometimes static makes itself known very clearly by the fact that, as we approach the trouble spot, our hair may actually stand on end or we may receive an unpleasant electrical shock.

In less obvious cases, a relationship between the intensity of the problem and changes in temperature and humidity may give a clue. If the trouble intensifies as the humidity goes down and the weather becomes cooler, suspect static.

If our textile problem involves a ballooning effect in yarn, or a repulsion of one yarn for another, or the attraction of yarn to a machine part, or if there is running out of guides, or if there is an unusual attraction by the fabric for lint or dirt, suspect static. Finally, any kind of problem in any dry operation involving one of the new synthetic fibers may be caused by static.

When we suspect static as the cause of a textile problem, however, we must make sure that static is really the cause and not something else. In some cases our suspicions may be partially substantiated by the use of an instrument recently put on the market. This instrument can measure or indicate an electric field, or a focal point of static electricity, when directed to the suspected place. Actually, however, if we suspect that static is the cause of trouble the only real positive proof is to treat the textile with the proper antistatic agent or to place static eliminators at the trouble spot and then to see if the trouble stops or is reduced. The odds are that we shall get this proof, for, when static is suspected there is a mighty good chance that it is there.

XV. *How do we choose an antistatic agent for a particular problem?*

While we may not have an ideal product to control static, we certainly can make the best of what we have. This selection involves a careful evaluation of our problem from many angles.

Suppose, for example, we are interested in the control of static in the sizing and weaving of undyed acetate warps. Here we are concerned not only with antistatic properties, but we must also consider the compatibility of the agent with our size mix. We may not be concerned with the effect of the agent on the lightfastness of colors or with its durability. The antistatic agent, however, must not have any harmful effect on subsequent processing. If, at the same time, the antistatic agent has some softening or lubricating properties, then this certainly would be a desirable property. Finally, the antistatic properties of the agent must be sufficient to remove the static that causes our particular problem.

Suppose, however, we were going to use an antistatic agent to control the development of static in a nurse's uniform, made of one of the newer synthetic fibers. Here, of course, the highest possible antistatic effect would also be an important property, but there would probably be no requirement for compatibility of the agent with other substances. Softening properties might be desirable, but not important. The color of the antistatic agent, however, would be important. No yellowing would be desirable and, finally, permanence, or at least renewability, of the antistatic agent would be of utmost desirability.

Take the matter of substantivity. It might be necessary to apply an antistatic agent to yarn in the form of skeins or packages for best results. Here, substantivity would be important, so that the agent could be applied economically and evenly to the yarn.

These examples are just a few of those we may actually encounter. Each of us must determine the requirements that an antistatic agent should meet for his own particular problem, such as compatibility, effect on color, effect on appearance of fabric, etc. This information may be supplied by the chemical manufacturer or be determined from preliminary laboratory

screening tests or be obtained from both sources.

Finally, after we have made our selection, we must apply these agents under actual plant conditions and note the practical results. If antistatic effects are needed from a manufacturing standpoint, the results will be obvious. The probelm is either eliminated by the antistatic agent or it isn't.

If the antistatic problem relates to consumer use, however, we face difficulties because the results are less obvious. Until such time as satisfactory laboratory testing instruments for antistatic properties become available, there is no answer other than to test a textile thoroughly under actual consumer conditions. Only in this way can we get the final story.

REFERENCE

(*1*). "Static Problems and their Control in the Textile Industry," Lopez and Hewson, *Am Dyestuff Reptr* 41, P105 (1952).

1952 Outing Photos—Rhode Island Section—Wannamoisett C C

Photos by John E Trezise

Patents—

(Concluded from Page 508)

fabric but persists on the printed areas only after washing. Several combinations with transparentizing or overdyeing aftertreatments are within the scope of this invention.

Example: Ponsol Jade Green (C I # 1101) is first vatted as usual and then added to dilute acetic acid. The obtained dispersion is incorporated into an aqueous paste containing Karaya Gum, Aerotex M-3, ammonium thiocyanate and Carbowax 1500. Fabrics printed with this paste are dried to a moisture content of about 10% and frictionated at 350° F. If the resin should not be completely polymerized in the hot calendering procedure, short aftercuring in a conventional chamber is suggested.

The inventors explain the fastness to rubbing of these prints by the "molecular or semimolecular" state of the vat acid dye pigment, penetrating the fabric and strongly adhering to the fibers. It would not be possible to reoxidize the vat in this process before calendering because it would cause smearing of the pattern. The cured fabrics can be reoxidized by passing through a peroxide solution as usual, but it has been found that this step is generally not necessary because satisfactory reoxidization chiefly occurs by the normal contact with air during printing, drying and curing.

Among other references cited by the Patent Office:

U S Pat 2,267,609 (National Aniline & Chem Co/1941): pasting vat dyes, such as National Vat Brown G or Indigo with an alkali- and water-soluble ester of an aliphatic monocarboxylic acid, such as sodium isobutyl sulfoacetate.

U S Pat 2,169,546 (Ciba/1939): dyeing or printing fabrics with acid, basic, direct or Indigosol dyes, using melamine-formaldehyde condensates as fixing agents.

U S Pat 2,123,153 (Celanese/1938): padding a fabric with dyestuff having practically no affinity to the fibers, overprinting with a nitrogenous resin solution (urea-formaldehyde), aging, and thereby fixing the dyestuff on the printed areas only.

U S Pat 1,909,221 (General Aniline Works/1933): condensates of urea and the ammonia compound of an aliphatic aldehyde, e g, the triazine type condensate of urea, and acetaldehyde-ammonia are added to a potash-Rongalite printing paste of vat dyes, especially of the anthraquinoid series.

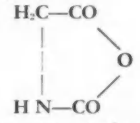
It is further suggested that this digest be compared with U S Pat 2,590,213 (Sandor) (see page 506), which describes dyeing with concentrated vat acid pastes.

DYEING SUPERPOLYMER FIBERS, Condensed with Amino Acid Carboanhydrides C, 4, 07

U S Pat 2,592,473
(Du Pont——Schneider——April 8, 1952)

This invention aims at improving the dyeing properties and, generally, the wetability of superpolymer fibers of the nylon type. The preamble to the specification refers to an older method comprising a reaction with formaldehyde and an alcohol, which results in the formation of N-alkoxymethyl polyamides.

Another method of modifying the fiber's substance to improve the dyeability is protected by the present patent. The superpolymers are characterized by the units —CONH—, separated by carbon chains of at least 4 C-atoms. They are treated with a carboanhydride of an amino acid. The simplest compound in this process is glycin-N-carboanhydride (also termed anhydro carboxy glycin):



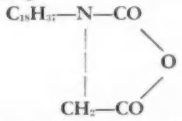
The inventor assumes that the amino-hydrogen groups present in the preformed polyamide react with the carboanhydride ring, causing scission of the ring and evolving CO_2. Thus the modified polyamides will contain a certain amount of amino acid residues.

Other reactive carboanhydrides include the products obtained by substituting for HN lower alkyls up to C_2, e g, sarcosine carboanhydride. Substitution compounds containing higher alkyl chains have a neutral or adverse effect: the final polyamide becomes less water sensitive, more water repellent and consequently less dyeable.

The carboanhydrides are condensed with clean, absolutely dry polyamide yarns in a solvent solution (chlorobenzene). The treated yarn is said to be slightly stiffer, but with no surface film formation. The yarns, dyed with Polar Red G Conc (C I # 430) or Cyananthrol R (C I #1076) gave brilliant deep shades while untreated yarns were only faintly colored under the same dyeing conditions.

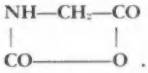
Among other references cited by the Patent Office:

U S Pat 2,327,162 (Imperial Chemical Industries/1943): high substituted diketo oxazolidines, e g,



from organic solutions, are used as water repellent agents.

Brit P 567,501 (Imperial Chemical Industries/1945): preventing felting of wool by a treatment with anhydrocarboxyglycin:



The first reference proves that products of the carboanhydride class, N-substituted by long chains, have water repellent properties as mentioned in the present specification. The second reference discloses that anhydrocarboxyglycin has antifelting properties. It is not to be concluded from either of these references that the same product could modify the wettability of superpolyamides.

ABSTRACTS

New Blends Create Problems for Dry Cleaners

(From a letter written by Paul Marcus, President of the New York Neighborhood Cleansers Association to the Federal Trade Commission.)

Textile Inds 116, 91, June, 1952.

The continued and increasing number of unfortunate and costly experiences which the members of the New York Neighborhood Cleansers Association are experiencing in endeavoring to dry clean apparel made of new and untried blends of man-made fibers with natural vegetable or animal fibers convinces this Association that more adequate protection is needed by retail merchants, by the purchasing public, and last——but not least——by the dry cleaning and laundering industries of this country.

It is becoming a more and more common practice to imitate fabrics of proven serviceability by substituting cheaper fibers or blends of fibers, cheaper dyeing processes, and chemical finishes. The result is that dry cleaners, judging the fabric by its external appearance, find that it will not withstand ordinary cleaning techniques which were used successfully on the original fabrics.

Ignorance or disregard on the part of manufacturers is resulting in the production of new fabrics which cannot be cleaned by any existing process. The unserviceability may be due to innovations of fibers or yarns; in the weaving; in the dyeing, or in the finishing. The public should be told of the service limitations of these fabrics. The dry cleaners should be told if they

can be cleaned and by what process.

New synthetic fibers are being marketed prematurely without having behind them any history of proven service. These new fibers are used in fabrics alone or in blends with animal or vegetable fibers. Some of these blended fabrics have characteristics which may be of great service to the public, but many of them simply cannot be dry cleaned.

In the cellulose fiber field, rayon is being widely used alone or in blends to make fabrics which were formerly made successfully from acetate, silk, or nylon. In using rayon, many of these fabrics after being woven are stretched and finished with water-soluble sizing. Mere drops of moisture——raindrops or a spilled drink ——dissolve the sizing. The dry cleaners must use moisture on any garment in order to dissolve any water-soluble stains such as sugar, liquor stains, etc. Perspiration odors can only be removed by water. There is no known method by which the dry cleaner can possibly clean these garments satisfactorily.

The writer requests the Federal Trade Commission to make a thorough investigation of present conditions and call a Trade Practice Conference to establish fair trade practice rules requiring that all garments made from these new fabrics have sewn to them suitable descriptive labels.

Properties of Apparel Wools. II. Modification of Fiber Surface During Worsted Processing

D F O'Reilly, J C Whitwell, R O Steele, and J H Wakelin, *Textile Research J* 22, 441-7, July, 1952.

Modification of the fiber surface during processing has been studied on three wools of Australian fine, domestic fine, and domestic medium grades. Samples were taken from each wool at each stage of their manufacture into a worsted fabric by the Forstmann Woolen Co. Groups of fibers, selected randomly from each sample, were treated with Pauly reagent and examined microscopically. The amount of color on the fiber was estimated visually, and the amount of scale disturbance was assessed. The rate of exhaustion of Anthraquinone Blue SKY was also measured on samples from each processing stage.

A consistently increasing amount of surface modification was observed with all three wools as the manufacturing process advanced. The susceptibility of the wools to staining with Pauly reagent and their rate of taking up Blue SKY from a dye bath also increased. However, all of these changes were reversed by a steaming process such as is frequently used in normal worsted processing.

Conclusions: The processing of wool produces surface modifications which are visible under the optical microscope. Steaming of processed wools reverses the surface modification to such a degree that the steamed samples nearly approach the condition of unprocessed wool. The surface modifications produced by processing may be evaluated by partial dyeing or by dyebath exhaustion methods. The dye susceptibility of processed wools is related to the amount of scale disturbance of the fibers, and the epicuticle does not appear to be the only major factor in resisting dye penetration.

A New Method for the Dyeing of Mineral Khaki

E H Daruwalla and G M Nabar, *J Soc Dyers Colourists* 68, 168-70, May, 1952.

The authors state that in the normal khaki dyeing process the textile is impregnated with a mixture of aqueous solutions of chromium sulfate and ferrous sulfate, dried, and then passed through a boiling solution of a suitable alkali to precipitate the metals as hydroxides. The process is lengthy, and variations in the conditions of drying, developing, and subsequent airing have been found to have a profound effect upon the resultant shade and fastness properties.

It is known that the presence of certain nonelectrolytes, particularly those containing hydroxyl groups, e g, cane sugar, glucose, glycerol, etc, can prevent the precipitation of the hydroxides of a number of metals on the addition of caustic soda. The solutions thus obtained remain clear and stable in presence of an excess of caustic soda. The precipitation of the hydroxides can later be effected at a controlled rate. The new continuous single bath process for the dyeing of mineral khaki on cellulosic textiles which is described by the authors is based on this principle.

The process consists in mixing the salts of chromium and iron with caustic soda in presence of glycerol to obtain a clear solution. The fabric is then impregnated with this solution, to which a coagulating agent is added which does not operate in the cold. The coagulation of the colloid, resulting in the precipitation of the hydroxides, is brought about by steaming the dried impregnated cloth. Dyeings having very satisfactory fastness properties are said to be obtained.

The padding liquor contains sodium dichromate mixed with sulfuric acid and reduced with sugar; ferrous sulfate; glycerol; caustic soda; and ammonium acetate. The fabric is padded with this solution, dried in hot air, steamed in an acid ager, washed, and soaped at the boil.

A large number of khaki shades are possible by variation in the concentration of the two metal components of the padding liquor. It is found that the shades produced as described above are greener than those obtained by the older process when the same proportions of iron and chromium are used.

Exposure tests show that the deposited oxides exercise a marked protective influence against the degrading action of light and air.

The authors state that the mineral khaki dyeings produced by the new process compare very favorably with those obtained by older methods, and that the method possesses other advantages, particularly in the simplicity of operation and the uniformity of the shades produced.

U S Liner Fireproof Throughout

Anon, *Dyer* 107, 963, June 27, 1952.

Because the specification of the new American liner *United States* required that the ship be completely fireproof, months were spent in research and experiment to find fabrics for curtains, bedspreads, etc, that were not only lovely to look at but also noninflammable.

Miss Urquhart and Miss Marckwald, partners of Smyth, Urquhart and Marckwald, New York, responsible for the interior *décor*, solved the problem by using a fire retardant, Pyroset Finish, for some of the fabrics, and by the extensive use of Dynel, a noninflammable fiber, for bedspreads and curtains.

Pyroset Finish was developed by the American Cyanamid Co. It is said to be a durable fire retardant which does not ruin color or texture, does not migrate, does not harm the fabric strength, and can be applied without special finish.

Application is simple. Pyroset is combined with phosphoric acid in an aqueous solution, the fabric to be treated is immersed in this, passed through a mangle, and then dried and cured. It is this curing operation (approximately 300°F for one minute) that gives the treatment the durability to withstand 25 wet or dry cleanings.

Dynel is a vinyl-chloride-acrylonitrile developed by the Union Carbide and Carbon Corp. The *United States* will carry 2,116 Dynel bedspreads and 598 pairs of curtains. The total yardage of Dynel is over 27,000.

This fiber, it is claimed, will last for years and is particularly easy to care for. It can be washed or dry cleaned without shrinkage (the texture is actually improved by washing) and the color-fastness is excellent, according to the manufacturers. It is proof against moths and mildew, an important factor at sea where dampness is always present.

NEWS

R A Hoekelman

AMERICAN CYANAMID COMPANY announced on August 1 the appointment of R A HOEKELMAN as assistant general manager of the Plastics and Resins Division.

Mr Hoekelman joined Cyanamid in 1930, and served in the accounting and credit departments until 1934, when he resigned to become associated with Maywood Chemical Works. In 1941 he rejoined Cyanamid where in recent years he has served as assistant treasurer and comptroller.

Prior to his association with Cyanamid, Mr Hoekelman was employed in the banking field and with A Klipstein & Company, chemicals and dyestuffs distributors.

ERNEST NATHAN has announced his resignation as vice-president of the Sun Chemical Corporation and chairman of the chemicals group comprising the Warwick Wax subsidiary and Warwick Chemical Division of Sun Chemical.

JARVID JONSSON has been appointed to the newly-created position of director of personnel administration for the Carpet Division of Alexander Smith, Inc, according to announcement by H S Worthington, vice president, operations.

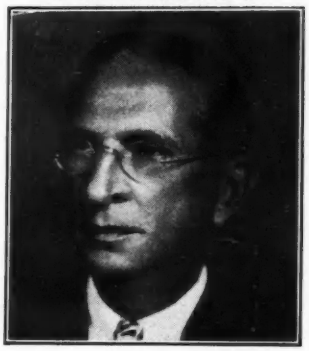
In the new position, Mr Jonsson will have complete responsibility for all phases of personnel policies, practices and administration.

Mr Jonsson was formerly with the Rohm and Haas Company, Philadelphia. Prior to joining Rohm and Haas in 1944, he was assistant to the vice president of manufacturing at Eavenson and Levering, Camden, N J.

JAMES C STRETCH, who for the past several years has been in charge of the accounting activities at the Rohm & Haas Company plant in Bristol, Pa, was elected assistant treasurer by directors of Rohm & Haas on July 22.

Mr Stretch joined the organization in 1942.

OBITUARY



LAMMOT DU PONT

LAMMOT DU PONT, 71, former president of E I du Pont de Nemours & Co, Inc, died in Wilmington on July 24th. He had been confined to his summer home at Fishers Island, N Y, for several weeks.

He was the youngest of the three brothers who had led the twentieth century development of Du Pont and was the eighth member of the du Pont family and the third brother in succession to become head of the firm. He served as president from 1926 to 1940 when he resigned to succeed his brother, Pierre, as chairman of the board.

JAMES R MORRISON

FUNERAL services for James R Morrison, 53, vice president, treasurer and director of the Morrison Machine Company, Paterson, N J, were held at the Van Emburgh Mortuary, Ridgewood, N J, on July 28th. Mr Morrison had been a member of the firm since 1918.

Surviving are his widow, Doris A; a daughter, Anne E; his father, James L, founder of the Company; a brother, John C, a vice-president of the Company; and a sister.

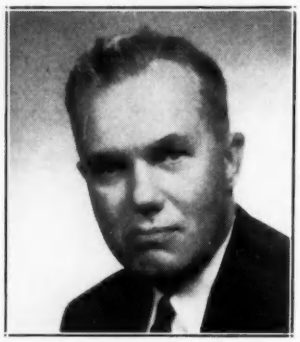
J P Madden

JOSEPH P MADDEN, assistant sales manager of the Stauffer Chemical Company, New York, has resigned to become vice-president and a director of the Caldwell Chemical Company, Inc, effective August 1. Caldwell is a manufacturers' representative and distributor for chemical products, with offices in New York and Detroit and Portland, Oregon.

Mr Madden joined Stauffer nineteen years ago as a member of the accounting department and was transferred to the sales staff in 1933. Seven years later he was named head of the Philadelphia sales territory, a post he held until 1942, when he was brought back to the New York office as assistant sales manager in charge of alkalies, chlorine and chlorinated products.

PRESTON FURMAN, 64, of Clifton, New Jersey, was honored recently by Heyden Chemical Corporation executives upon completion of 50 years of continuous service with the company, including 32 years in the Heyden Sales Department, covering the state of New Jersey and other territories.

Mr Furman was presented with a thousand dollar coupon-type U S government bond by John P Remensnyder, president of Heyden, and was elected the first member of the Heyden Fifty-Year Club at a luncheon held in his honor by Heyden executives. Executives of leading chemical companies and chemical trade associations sent wires and letters congratulating Mr Furman.



H A Sweet

Mr Sweet was recently named director of the Industrial Division of Refined Products Corporation, Lyndhurst, N J. (See page 500 of the August 4 issue.)

AMOS K HOBBY has resumed his position as chemist in the Chemical Research Laboratory of the Woonsocket Color & Chemical Company where he will lend additional impetus to the research program currently being undertaken.

Mr Hobby was formerly associated with Merck & Co, Rahway, N J, Onyx Oil & Chemical Company, Jersey City, N J, and Anglo Fabrics.

THE Dow Chemical Company announced on July 21st the appointment of RAYMOND F BOYER, former director of the company's physical research laboratory, as manager of plastics and high polymer research.

Mr Boyer will be succeeded by DR WILLIAM C BAUMAN, assistant director of the laboratory since 1948, and will replace L C CHAMBERLAIN, who has been named assistant to the director of research, DR R H BOUNDY.

In 1935, Mr Boyer went to work for Dow in the physical research laboratory, where he specialized in the physical chemistry of plastics. He advanced to assistant director of the laboratory in 1945 and director in 1948.

Mr Chamberlain joined the physical research laboratory in 1928. He became assistant director of the laboratory in 1941 and four years later he was named head of plastics research. He has been a key figure in numerous Dow process developments, including iodine, bromine from sea water, and the Dowell process for acidizing oil wells.

Dr Bauman entered Dow's physical research laboratory in 1938. He became assistant director of the laboratory ten years later. After early work resulting in improved processes for the recovery of iodine, he started on the development of ion exchange resins.

GRAVER Water Conditioning Co, New York, manufacturers of equipment for all water-conditioning processes, is further expanding its technical organization to extend its services to central power generating stations, industrial plants and municipalities.

A new Research and Product Development Department has been created under the direction of VINCENT J CALISE, who has developed many of the Company's equipment designs since its establishment as a division of Graver Tank & Mfg Co, Inc, and has been associated with recent advances in the field of water conditioning.

MARVIN LANE, who has been advanced to the position of technical manager, has specialized for many years in water conditioning laboratory, pilot plant, field experimental and plant operating work, and has been the Company's chief chemist since 1949. He has also had extensive experience in the development of new ion exchangers and in problems relating to demineralizing, silica removal, and water softening and clarification. In his new capacity, Mr Lane's responsibilities will include design development, process standards and guarantees, water treatment recommendations, and analysis of installation performance.

The Company's Customer-Service Department will be in charge of WILLIAM J LEWIS, who has had considerable research, field engineering, plant operation and installation servicing experience in water conditioning equipment. Mr Lewis' first connection with Graver was in 1941 as Field Engineer for Graver Tank and Mfg Co, Inc, and he joined the Service Department of the Water Conditioning Co in 1950.

PITTSBURGH COKE & CHEMICAL COMPANY has announced the addition of BOYCE C BOND, as sales supervisor, and KARL C RUPPENTHAL, as technical supervisor of the firm's newly-formed Fine Chemicals Division.

Mr Bond, who was associated with General Dyestuff Corporation for 19 years, recently developed and patented the Bond Machine, a new universal type continuous dyeing machine. In his new post he will serve the textile trade in both a technical and sales capacity. His office will be at Kenilworth Road, Villanova, Pa.

Mr Ruppenthal, who will be in charge of the modern sales service laboratories which will service the range of dyestuffs and textile auxiliaries to be offered by PC&C, was formerly a technical demonstrator and textile chemist in charge of the Philadelphia laboratory of General Dyestuff Corporation. He has had 13 years' experience in dyestuff application and the leather trade.

W B Reese

MACBETH CORPORATION and subsidiaries, Newburgh N Y, have announced the appointment of WARREN B REESE as general sales manager.

Mr Reese joined Macbeth in 1950 and since has been occupied with industrial color problems through applied color-corrected illumination and color measuring instruments. His experience includes research on basic color problems and the development of industrial instruments and lighting equipment for industrial color control.

ALLEN R WILLIAMS, one of the senior members of Du Pont's Textile Service Section at 44 Worth St, New York, N Y, has retired after twenty-eight years with the company.

Born in Amesbury, Mass, Mr Williams attended Lowell Textile Institute and began his textile career in 1908 as an apprentice in the Hamilton Cotton Mills in Lowell, Mass. A first lieutenant in the U S Army during World War I, Mr Williams in 1919 became a salesman in the Boston office of I Levinstein & Company and Dyestuffs Corporation of America (later acquired by Du Pont).

In 1936 he was transferred to Du Pont's Providence, R I, dyestuffs office and in 1937 was appointed a member of the Textile Service Section in New York, his most recent post.

FOUR appointments have been made to the Textile Fibers Department of Carbide and Carbon Chemicals Company. They include RICHARD S BRISSETTE, HUGH L CAROLAN, ROGER A LEWIS and ROBERT A SIMMONS.

Mr Brissette will be a technical representative for textile fibers in the New England area. Mr Carolan will be engaged in the company's fiber advertising and promotion activities in the New York office. Mr Lewis has joined the Dynel Production and Operating Department at South Charleston, W Va; and Mr Simmons will be associated with the Development Service Laboratory at South Charleston, W Va.

H S Mellin

HELEN S MELLIN, president and treasurer of Mellin Chemicals, Inc, Ridgewood, N J, is one of the newest members of the American Association of Textile Chemists and Colorists, having been granted an Associate membership.

Mrs Mellin is carrying on in a business inaugurated by her late husband, Nelson F Mellin.

THE FOXBORO COMPANY, Foxboro, Mass, has named JOHN DEADERICK manager of the Philadelphia office, which services eastern Pennsylvania and southern New Jersey.

JOSEPH ESHERICK, for many years district representative for Foxboro in Philadelphia, now devotes his entire attention to Special Accounts, serving as manager of that department.

Mr Deaderick has had extensive experience in the oil and gas industries, previously serving as industrial engineer at Foxboro branches in Corpus Christi, Texas, and Baton Rouge, La, and as manager of the Tulsa, Oklahoma branch.

CRAWFORD H GREENEWALT, president of E I du Pont de Nemours & Co, Inc, Wilmington, Delaware, has been chosen to be the nineteenth recipient of the Chemical Industry Medal according to a recent announcement by the American Section of the Society of Chemical Industry, donor of the medal. The medal will be formally presented to Mr Greenewalt at a meeting of the American Section following a dinner in the medallist's honor at the Starlight Roof of the Waldorf-Astoria Hotel, New York, N Y, on October 31.

The Chemical Industry Medal was established in 1933 and is awarded annually in recognition of conspicuous services to applied chemistry. Mr Greenewalt was elected to receive the medal by the Executive Committee of the American Section of the Society of Chemical Industry sitting as the committee of award.

Harry B McClure, vice president of the Carbide & Carbon Chemicals Company, and chairman of the American Section, will preside at the award meeting and Dr Robert C Swain, vice president of American Cyanamid Company, and immediate past chairman of the American Section, will present the medal.

Mr Greenewalt has been president, chairman of the Executive Committee, and a member of the Finance Committee of E I du Pont de Nemours & Company since January 19, 1948, the peak in a career begun in 1922 when he joined the Philadelphia Works of the company as a chemist. He is the tenth president of the Du Pont Company, and the second man not of the du Pont name to occupy the presidency in the 150 years of the company's history. Before assuming the presidency, Mr Greenewalt had been stationed in four manufacturing and two auxiliary departments, and had been a vice-president and a director of the company.

A native of Cummington, Mass, Mr Greenewalt is a graduate of William Penn Charter School in Philadelphia and Massachusetts Institute of Technology, where he received a bachelor of science degree in chemical engineering in 1922. He joined the Du Pont Company immediately after graduation as control chemist in the heavy chemicals laboratory in the Philadelphia Works of the Paint, Lacquer and Heavy Chemicals Department.

W F REICH, JR, Vice-President of Carbide and Carbon Chemicals Company, a Division of Union Carbide and Carbon Corporation, has announced the appointment of R F BROWN as an assistant sales manager in the Company's Industrial Chemicals Division.

Mr Brown was formerly Midwestern Division Manager with headquarters in Chicago and he will move to New York shortly to assume his new duties. His position in Chicago will be taken by R M JOSLIN, JR, who moves up from District Manager of the Philadelphia area.

The Industrial Chemicals Division handles only those chemicals produced by the Company for which markets have already been established and are sold in drum, tank car, or tank truck shipments. This Division is headed by N C BABCOCK, who is assisted by H D HUGHES, sales manager, and five assistant sales managers, R E DALEY, E E FOGLE, at present with the NPA in Washington, C J HERRLY, R B LEONARD and Mr Brown.

NEW PRODUCTS AND DEVELOPMENTS

• Emery Unveils New High-Quality Worsted Oil

Emery Industries, Inc, Cincinnati, has released details on Twitchell 7421 Worsted Oil, a new high-quality worsted oil with antistatic properties. Extensive, continuous mill tests have reportedly established its utility as a fiber lubricant.

Important features of 7421 Worsted Oil, as quoted by Emery, are:

Static Control: "provides the static control required by high-speed processing in most instances. This is true for integrated worsted operations where this oil is applied throughout, or where tops are processed with at least 1.5% oil."

Emulsion Fluidity: "Water emulsions containing as high as one part of oil to one part of water are still fluid enough to be used in fine spray application systems. This permits the use of high oil concentrations without using excess water or creating stickiness or gumming."

Emulsion Stability: "The exceptional emulsion stability of this product is evidenced by the absence of oil separation in water emulsions after standing for several weeks. This is true of emulsions ranging from 1 part oil to 9 parts water up to as high as 1 part oil to 1 part water. In addition, the penetration and spreading properties of these emulsions are excellent."

Resistance to Yellowing and Odor Development: "Of considerable importance to high quality worsteds is the outstanding ability of Twitchell Oil 7421 to resist yellowing, staining and the development of rancidity and other odors upon aging. This is very essential to dry finished fabrics where any staining, yellowing, or dulling of colors is reflected by decreased sales-appeal."

Excellent Scourability: "In normal plant scouring, even after prolonged aging, an average residual oil content of 0.3% is easily obtained. This excellent scourability compares with that of the self-scouring types. Soap costs are minimized."

Rust Preventitive: "Water emulsions of Twitchell 7421 are very effective inhibitors of corrosion and rust."

A descriptive technical bulletin covering this product is available on request from: Emery Industries, Inc, Dept 5, Carew Tower, Cincinnati 2, Ohio.

• New Ciba Products

Ciba Company, Inc has announced the availability of a new dyeing assistant, a new black, and three additions to the Ciba Neolan range of dyestuffs.

Dyeing Assistant #52 is recommended by the manufacturer as a carrier or dyeing assistant in the application of the Cibacete colors on Dacron. It is marketed in liquid form which can reportedly be diluted with water and added directly to the dyebath. Pre-dissolving with caustic soda and the subsequent addition of acetic acid to the dyebath are not required, it is stated. The product is said to leave Dacron with a soft, lofty hand.

Neosol Printing Black BD is said to produce a comparatively low cost black with excellent fastness on all cellulosic fiber materials, distinguished by a bloomy deep shade and sharply-defined details. Because of its resistance to hydrosulfite, this dyestuff is especially recommended by Ciba for producing fine outlines, free from halos, alongside vat color and white discharges. Also, it is claimed very useful when used as a black on white or colored overfalls.

Neolan Green 8G, Neolan Green BF, and *Neolan Flavine GFE* have been developed to brighten the range of greens and yellows heretofore available. They are said to dye level, to be simple to apply and easy to shade.

Data and samples of each product are available on request.

• Woonsocket Introduces New Permanent Duller

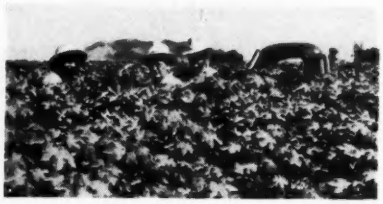
A new permanent duller for viscose, acetate rayon and cotton has been introduced to the trade by the Woonsocket Color & Chemical Company, Woonsocket, R I.

The new product is said to be compatible with urea-formaldehyde and melamine resin after-finishes, starches, gums, etc. It has been described as a polyresinous nonionic compound.

• PC&C Division to Manufacture Systemic Insecticide

The Pittsburgh Agricultural Chemical Company, a division of Pittsburgh Coke & Chemical Company, will manufacture Systox, an emulsifiable spray concentrate for the control of aphid and mite on cotton. The new product is reportedly the first systemic insecticide ever approved for use in this country.

Extensive large-scale field tests last season are said to have revealed that probably only two applications will be required per season to give complete control of these two insect pests. The two-spotted mite is of major economical importance in the cotton-producing areas of Southern



An average field of cotton showing the bush growth and vigor of "Systox"-protected plants.

California and Arizona.

The new insecticide is an organic phosphate ester. After being applied either to the foilage or to the soil, it is absorbed and spreads throughout a treated plant by a physiological action known as translocation, making the entire plant highly toxic to many destructive insects for a considerable period of time. Even new growth is said to be protected through translocation.

• Aquex BNWC Employed in New Permanent Rayon Finish

A new permanent finishing process that, it is claimed, renders 100% spun viscose rayon fabrics unconditionally washable, has been developed by the Aquex Development & Sales Corp, Whippany, N J.

The process employs Aquex BNWC—one of the finishing resins recently developed by the Aquex Company—on conventional finishing equipment. E W Sweet, vice president and general manager, reports that actual mill production runs have shown that fabrics treated with the new process have the advantage of low residual shrinkage when washed at the boil and no progressive shrinkage, even when washed many times at the boil.

The same tests reportedly demonstrate that fabrics treated with this process are resistant to the damaging effects of chlorine, acid and alkali washes. Such fabrics can be bleached in the home or in commercial laundries without any danger of yellowing or any loss of fabric tensile strength, it is claimed. Another advantage is the improvement in the wet strength of the fabric.

Practically all types of hand can be obtained with this process, according to Mr Sweet, from that of a soft shirting to the hand of a full dress fabric.

Aquex officials state that the new finish should find a place in the finishing of rayon fabrics where the primary requisite is complete, unconditional washability .

• Improved Titrimeter Speeds Karl Fischer, Micro, Other Titrations

As Fisher Scientific Company, Pittsburgh, Pa celebrates its Golden Anniversary, Company development engineers have announced a new improved Titrimeter. This electronic instrument for the quantitative determination of solutions features a completely new stand; improved stirrer and control box; and integral provisions for micro-titrations and Karl Fischer moisture determinations.

The stand is a massive, functional casting which supports the burettes and sample beaker so that they are close to the magnetic stirrer, electrodes, and electronic end-point indicator. It has a hood to protect all connections from "hand capacity" effects.

Magnetic stirrers and fixed electrodes serve to eliminate all shafts and belts; there is no oil or dirt to drop into the sample and stirring is simplified in such closed systems as are used in Karl Fischer work.

The motor, now mounted on a swivel head right under the beaker and stirring magnet, was selected to give a counter-clockwise stirring. The stirrer sweeps the entering reagent towards the sensitive electrode, furnishing—when the burette is properly positioned—a preliminary blinking of the electronic eye, which forewarns the operator of the approach of an end-point.

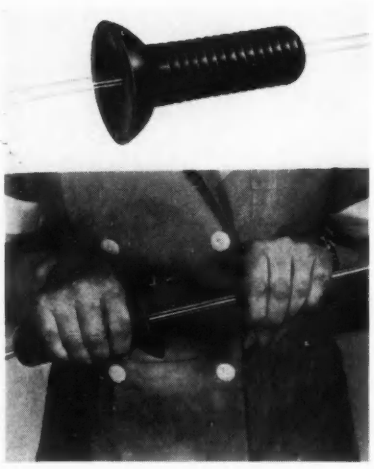
Stirring speed can now be set at any point, and once the desired stirring action is obtained, the operator need never reset the motor for any of the other sample beakers in his titration series, it is claimed, since a switch automatically interrupts the stirring action when the stirrer is swung outward and re-starts the stirring action when the stirrer and a new sample are returned.

In the Titrimeter's single, compact control unit are combined the means for amplifying the electrical potentials picked up at the electrodes and the means for detecting these changes via the end-point indicator eye. In addition, provision has been made for polarized electrode systems such as those used in Karl Fischer determinations. The dial is set at the "Pol" position and the platinum electrodes are used.

A calibrated dial and two controls are the only manipulative parts on the new control case. The scale on the dial is graduated in pH units as well as volts.

A special micro-titration accessory kit includes a micro glass electrode, a micro calomel electrode, a micro electrode holder, a micro magnet sealed in polyethylene, a micro burette and a 10-ml sample beaker.

With the Karl Fischer-Titrimeter setup, a laboratorian can run fast, accurate analyses of organic liquids, such as alcohols, ketones, esters, oils, fats, petroleum fractions; inorganic solids (Fuller's Earth, copper sulfate, calcium carbonate, water of hydration); and other miscellany.



Labline Grip-Safe

• New Tubing Manipulator Protects Hands

Long recognized as a dangerous procedure, the insertion of glass tubes and thermometers in stoppers has always been a frequent cause of accidents in the laboratory. Now the new Labline Grip-Safe, (Patent Pending) a product of Labline, Inc, 4710 West North Avenue, Chicago 39, Ill, reportedly eliminates this hazard by providing a gentle, but firm gripping action which permits sliding the tubing into stoppers without excessive or uneven pressure. In case of breakage, Grip-Safe is said to protect the hands, wrists and knuckles from injuries.

The new manipulator reportedly accommodates most sizes of commonly-used tubing as well as irregular shaped tubing, such as T's and L's. It may also be used to safely break tubing to desired length after marking with file or cutter, the manufacturer points out.

Grip-Safe is made of high grade Neoprene and is listed at two in a box for $5.95.

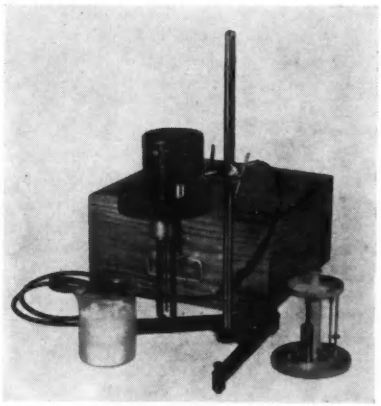
• 1952 Line of Brookfield Viscometers

Brookfield Engineering Laboratories, Inc, Stoughton, Mass, have announced the addition of several new models to their 1952 line of Brookfield Synchro-Lectric Viscometer's as well as a number of new features that have been added to all standard models of the Brookfield Viscometer.

Illustrated is the new eight-speed Brookfield Viscometer, designated as Model LVT, with a reported 200 to 1 ratio of speeds starting at .3 rpm. When equipped with a UL Adapter for special amplifying effects, the new Model LVT can measure any viscosity between .5 cps and 2,000,000 cps with extreme accuracy, it is claimed.

In addition to the eight-speed model and a new, modified four-speed model, the 1952 line includes some 10 other single and four-speed models in rpm's up to 600. All are portable and feature a new bubble level permanently attached for proper leveling either in a laboratory stand or when making point-of-process determinations. Any of the standard models may reportedly be secured with explosion-proof construction when desired, a modification that adds very little to the instrument's weight and bulk.

As shown, each new viscometer is accompanied by a set of suitable spindles, a spindle holder, and a carrying case which can contain both the viscometer and the spindle holder when they are not in use. Determinations are secured directly in centipoises by inserting the spindle in the material to be measured, flicking on a switch, and reading the dial.



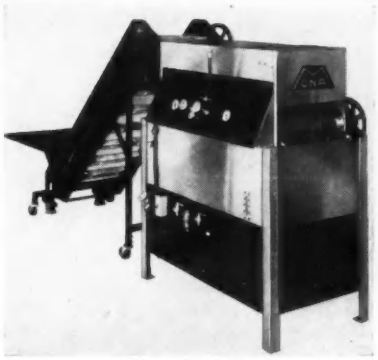
Eight-Speed Brookfield Viscometer

• Fuse-Din Resistance Thermometers

Weiller Instruments Corporation, 95 Broad Street, New York 4, N. Y, suggests that their Fuse-Din Resistance Thermometers, when used in association with Wheatstone Bridges, are excellent for reading temperatures ranging from —400°F to 3000°F.

To make the reading of temperatures convenient, resistance units are made with an ice point resistance of 100 ohms. With each bulb, a table is supplied which gives the ratio $K = \frac{R_t}{R_o}$ (R_t indicating resistance at the temperature to be measured and R_o the resistance at the ice point). If the unit has an ice point resistance of 100 ohms, the bridge will read direct—in K.

Fuse-Din Resistance Theremeters are reportedly made in Type BA, glass and pyrex; Type BD, the same with high speed tip; Type BR, the same, for extreme speed of response; Type MQ, moly in quartz; Type BM, metal clad, tungsten in pyrex; and Type MM, moly in quartz, metal clad.



Monalit-Duplex Conditioner

• Cold Water Yarn Conditioning Machine

The Monalit-Duplex, a new streamlined cold water yarn conditioning and twist-setting machine manufactured by Mona Industries, Inc, 65-75 East 23rd Street, Paterson 4, N J, finds increasing application for yarn on bobbins, quills, cones and other large packages.

For conditioning of bobbins and quills the yarn is fed into the machine by a conventional hopper and feeder conveyor unit. For cones and large packages, the mobile feeding unit is rolled aside so that the operator can feed the packages into the unit by hand.

The Monalit-Duplex is of stainless steel construction. All sides are enclosed with operating controls on the outside. The conventional wooden conditioning aprons have been replaced by stainless steel mesh conveyors, which permit spraying from both top and bottom. The machine is also said to be "drip-proof".

To facilitate servicing and cleaning, the unit is of a three-section light weight construction. The mobile feeding unit can be rolled aside, whereas the entire top section can be lifted off the lower tank and drive section for periodic cleaning of the tank and conditioning conveyor.

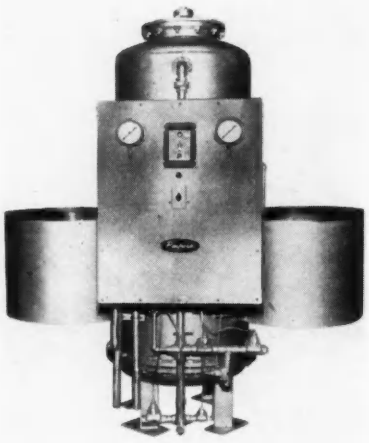
Sizes conform with individual mills' production requirements.

• Three New Antistatic Agents

Finetex Incorporated, Pompton Plains, N J, reports that three of their new antistatic agents, namely, Curdite XXR, Finestat 008, and Cordex 220, have been tested and approved by several leading manufacturers of synthetic yarns. Finetex states that all three in concentartions as low as 0.2 of 1% on yarn fiber and woven fabrics will completely eliminate static, will not bleed colors, or affect the shade and dye fastness of fabrics.

Heat stability is said to be excellent when the new products are applied to nylon, Dacron, Orlon, Saran and viscose.

Technical bulletins on each are available from Finetex.



Penfield Mono-Bed Demineralizer

• Latest Penfield Mono-Bed Demineralizer

The Penfield Manufacturing Company, Meriden, Conn, has added to its demineralizing equipment line a new, fully-automatic Mono-Bed Unit.

Whenever the effluent's conductivity falls below standard, the treated water is automatically discharged and lights (or other warning system) signal the need for activation of a regeneration cycle. The turning of a single switch then puts the unit through its regeneration cycle automatically, including rinsing and recutting in the effluent when desired resistivity (purity) is reached. During this time, the regenerative tanks are refilled with water so that fresh acid and caustics may be added in preparation for the next regeneration cycle.

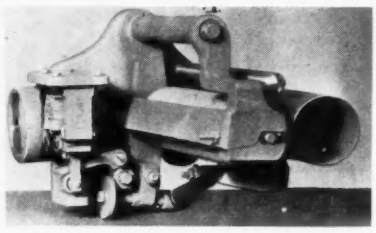
In the Penfield Mono-Bed Demineralizer cation and exceptionally strong anion resins are intimately mixed in a single unit tank through which the raw water passes only once.

Reportedly available in flow rates up to and beyond 5,000 gallons per hour, the new Penfield product is said to require no steam power, to operate for a chemical cost of only pennies per 1,000 gallons of treated water, and to provide an effluent with a mineral content of virtually zero (resistances are said to have been reported as high as 20,000,000 ohms per centimeter).

A complete catalog information on Penfield's full line of Mono-Bed and Multi-Bed Demineralizing equipment, may be secured by writing to Edward H Clohessy, Penfield's General Manager.

• Fast AC Electric Guider Operates in 1/50th Second

An electrically-operated guider which reportedly plugs into any 110-volt 50 or 60 cycle A-C line has been added to the line



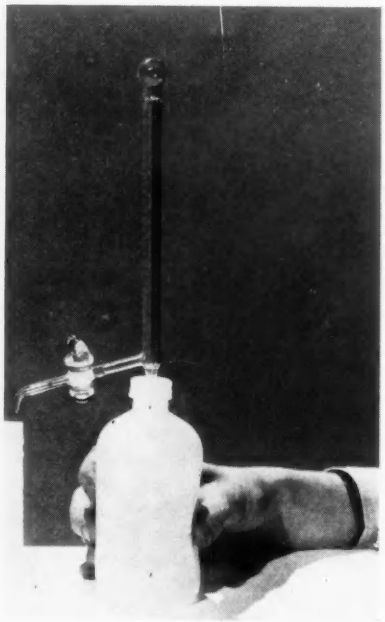
A-C Electric Guider

of air- and mechanically-operated guiders made by the Guider, Roll & Service Company, Daytona Beach, Fla, a concern that has heretofore made electric guiders operating only on direct current rectified from A-C. The new guider, according to the makers, opens and closes the metal and rubber rolls in 1/50 second.

Other features of the new guider include instantly-adjusted pressure between the metal and rubber rolls, quick adjustment of the distance that the rolls open, and operating switches that can be replaced in a few seconds. These switches are equipped with newly-developed arc suppressors, which, the makers claim, give them almost unlimited life. For extremely delicate fabric the new guiders can be furnished with electric eyes, which the fabrics' selvedge energize. The new A-C guiders fit on existing stands.

• New Automatic Burette

An automatic zero type burette, specially constructed for use with a polyethylene plastic reservoir has been well received by the trade following its initial appearance this past Spring.



Royalton Press-O-Matic Burette

Marketed as the Royalton Press-O-Matic Burette (Meyer Scientific Supply Co, Inc, 215 North 8th St, Brooklyn, N Y), the product requires only a gentle squeeze of the bottle to fill it. Chief advantages appear to be the greater safety against breakage as offered by a polyethylene delivery tube, and the simplicity of assembling——the burette is screwed into the reservoir.

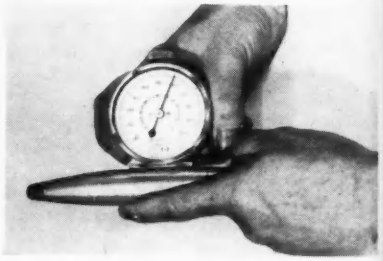
Available in 10 and 25 ml sizes complete with burette.

• New Instrument for Testing Winding Density

Aware of the fact that, to insure uniform unwinding in later processes, it is necessary that packages of yarn be wound to a uniform density, the Shore Instrument & Manufacturing Company, 90-35 Van Wyck Expressway, Jamaica 35, N Y, has created a precision testing instrument called the Durometer. The instrument is expressly designed for testing the density of windings on cones, spools, bobbins, cops and package dyeing tubes. Once the density is determined, a standard can be set for uniform winding.

The Durometer consists of a dial graduated in units from zero to 100. At the bottom, a blunt pin projects a standard distance. When pressed firmly against a test sample, the pin is forced back into the instrument in proportion to the density of the winding. The tighter the winding, the more it resists penetration of the pin and consequently, the higher the reading.

The pin is mounted on standardized twin leaf springs within the instrument. A pressure of two pounds is said to be required to deflect these springs to their maximum limit. On an inpenetrable test sample, this would yield a dial reading of 100, or slightly in excess thereof. The manufacturer points out that the ready adaptability of the Durometer to free hand testing permits tests to be made on bobbins that are attached to winding machines, etc. The instrument may be held in any position.



The Durometer